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INVESTIGATION OF STRUCTURAL PROPERTIES

OF FIBER GLASS FILAMENT-WOUND PRESSURES VESSELS

AT CRYOGENIC TEMPERATURES

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DOUGLAS

QUARTERLY PROGRESS REPORT

NUMBER THREE

INVESTIGATION OF STRUCTURAL PROPERTIES OF FIBER GLASS FILAMENT-WOUND PRESSURE VESSELS AT CRYOGENIC TEMPERATURES

APRIL, 1964

DOUGLAS REPORT SM-45893

PREPARED AND SUBMITTED BY: J. M. TOTH, JR.
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PREPARED FOR:
NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
LEWIS RESEARCH CENTER
CLEVELAND, OHIO
CONTRACT NO. NAS 3-2562

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for C. Y. Kam

APPROVED BY: H. H. DIXON
CHIEF, STRUCTURES BRANCH
ADVANCE SPACE TECHNOLOGY

DOUGLAS MISSILE & SPACE SYSTEMS DIVISION

QUARTERLY PROGRESS REPORT
NUMBER THREE

INVESTIGATION OF STRUCTURAL PROPERTIES OF FIBER GLASS FILAMENT-WOUND PRESSURE VESSELS AT CRYOGENIC TEMPERATURES

APRIL, 1964

SM-45810

Prepared for
National Aeronautics and Space Administration
Lewis Research Center
Cleveland, Ohio

Contract No. NAS 3-2562

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ABSTRACT

19619

This is an interim report on a development program for the investigation of structural properties of fiber glass filament-wound pressure vessels at cryogenic temperatures.

The following tasks were completed during the period of the report:

1. Liner evaluations were continued through the quarter by liquid hydrogen testing with the 7 1/2-inch diameter biaxial specimen. A nickel lined specimen was cycled 250 times at 36% of ultimate (600 psi); a glass flake-lined specimen leaked excessively at a pressure of 270 psi; a Mylar lined specimen leaked excessively at 360 psi; and an H-Film lined specimen leaked excessively at 355 psi. Another nickel lined specimen was cycled twice at 93% of ultimate (1540 psi).

2. On the basis of the results to date, the metals appear to show the most promise as integrally bonded liners. Due to its excellent cyclic behavior at 36% of ultimate (-423°F), most favorable differential contraction characteristics, and easiest and most satisfactory deposition process, electrodeposited nickel will be used as the liner for the 18-inch diameter pressure vessels.

3. Initial design of the 18-inch diameter pressure vessels has been completed.

4. Due to the selection of a metallic liner, considerable tooling development was necessary to satisfactorily seal the salt mandrel from the aqueous deposition bath. An acrylic laquer coating made by a repeated dip-coating operation appears to offer a solution to the problem. Retooling was also completed to rigidize the salt-steel mandrel interface to minimize flexural bearing-compression forces during seal coating, electrodeposition, and filament winding.

5. Fabrication, testing, and quality control procedures have been finalized for the 18-inch diameter vessels.

Author

NOTICES

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FOREWORD

This report was prepared by the Douglas Aircraft Company, Inc., Missile and Space Systems Division, under NASA Contract NAS 3-2562. This investigation was initiated by Lewis Research Center of NASA to determine the structural properties of fiber glass filament-wound pressure vessels at cryogenic temperatures. The work is being administered by Mr. James R. Barber, Chemical Rocket Systems Division, NASA/Lewis Research Center, 21000 Brookpark Road, Cleveland, Ohio.

This is the third quarterly progress report and covers work done between 1 January 1964 and 31 March 1964.

Included among those who cooperated in the research and the preparation of the present report were: J. M. Toth, Jr., Investigation Director, W. C. Loomis, and D. J. Soltysiak, Advance Space Technology; R. B. Lantz, Materials Research and Production Methods; R. T. Pfaffenberger, R&D Tooling; R. Yeaman, Engineering Research and Development Laboratories; and D. W. Yockey, Reliability.

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SECTION 1

INTRODUCTION

This document is the third quarterly progress report for Contract Number NAS 3-2562, sponsored by NASA/Lewis Research Center, Cleveland, Ohio. The project is a 16-month development program divided into four phases. The scheduling of the four phases has been arranged to permit the required "closed loop" between design, test, and fabrication.

The Phase I effort includes the orderly investigation and recommendation of liner materials and resin/fiber glass composites suitable for use with either liquid nitrogen or liquid hydrogen. Liner materials under investigation were Mylar, Tedlar, H-Film, polyurethane film, glass flakes, and electrochemically deposited nickel, silver, and copper. Three different resin systems were chosen for evaluation with S994 glass filaments.

Two 18-inch diameter by 24-inch long filament-wound pressure vessels will be designed and fabricated for testing with liquid hydrogen.

Twenty 18-inch diameter by 24-inch long pressure vessels will be fabricated in Phase II for testing with either liquid nitrogen or liquid hydrogen. The fabrication schedule will be paced by the testing schedule since there is a possibility of revising the vessel design, as the test program progresses, in order to provide an acceptable failure mode.

The Phase III effort consists of burst and cycling tests of small-scale fiber glass filament-wound pressure vessels at liquid nitrogen temperature. The Phase IV effort consists of burst and cycling tests of small-scale fiber glass filament-wound pressure vessels at liquid hydrogen temperature.

The objectives of Phases III and IV are:

1. Determination of burst strengths at -320°F and -423°F.
2. Determination of number of cycles required to fail the filament-wound pressure vessels at 60%, 70%, 80%, and 90% of ultimate pressure determined in (1) above.

3. Determination of the tank circumferential and longitudinal strains as a function of vessel pressure for each cycle.

4. Establishment of the efficiency parameter PV/W, using burst pressure for P and total vessel weight for W.

SECTION 2

PHASE I - LINER AND RESIN SYSTEM EVALUATION

In Phase I, the objective is to design and develop small-scale fiber glass filament-wound pressure vessels to contain cryogenic fluids. To reach this goal, Douglas is conducting an orderly investigation of liner materials and fiber glass resin composites for cryogenic applications. The materials and processes being used in the small-scale fiber glass tanks are also suitable for use in full-scale tanks.

During this phase, liner compatibility and material properties have been determined by means of test coupons and small cylinders. The properties of the liner and fiber glass include tensile yield and ultimate strength, modulus of elasticity, ultimate elongation, cyclic resistance, coefficient of thermal contraction and density. The permeability of the liner to gaseous nitrogen and hydrogen has also been determined. On the basis of information obtained during the previous two quarters (references 1 and 2) and this quarter, the following materials have been chosen, with the advice and approval of the NASA/Lewis Program Manager, for fabrication of the 18-inch diameter by 24-inch long filament-wound pressure vessels (two such vessels in Phase I):

Liner: 5 mil (0.005") electrodeposited nickel - Electroforms Inc. E510 Grade I

Resin System: Bakelite ERLA 0510/Bakelite ZZLO803.

Fiber Glass: Owens-Corning S994/HTS Fiberglas.

Adhesive System: Narmco 7343/7139.

2.1 LINER MATERIAL INVESTIGATION

All of the liner materials have been tested as coupons and results are reported in the First Quarterly Progress Report (reference 1).

2.1.1 Mechanical Properties Tests

All uniaxial coupon testing has been completed (reference 1).

2.1.2 Coefficient of Thermal Contraction

All coefficient of contraction testing has been completed (reference 1).

2.1.3 Cyclic Tests

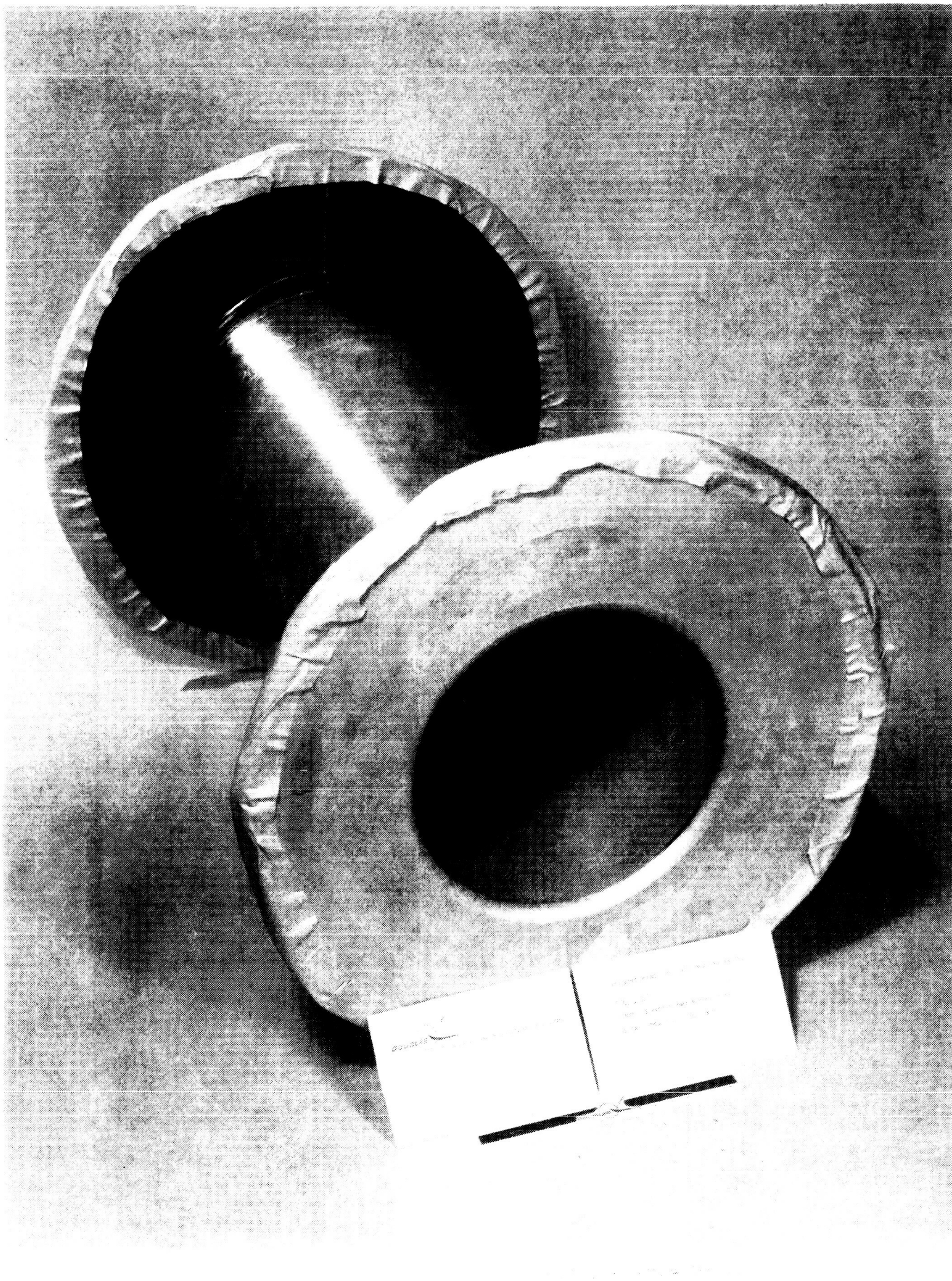
Cyclic testing with the 7 1/2-inch diameter biaxial test specimen was continued during the third quarter. Testing with this specimen provides for complete evaluation of a candidate liner material under stress at the desired cryogenic temperature, subject to representative internal pressures using liquid nitrogen or liquid hydrogen as the pressurizing medium. All testing to date has been with liquid hydrogen.

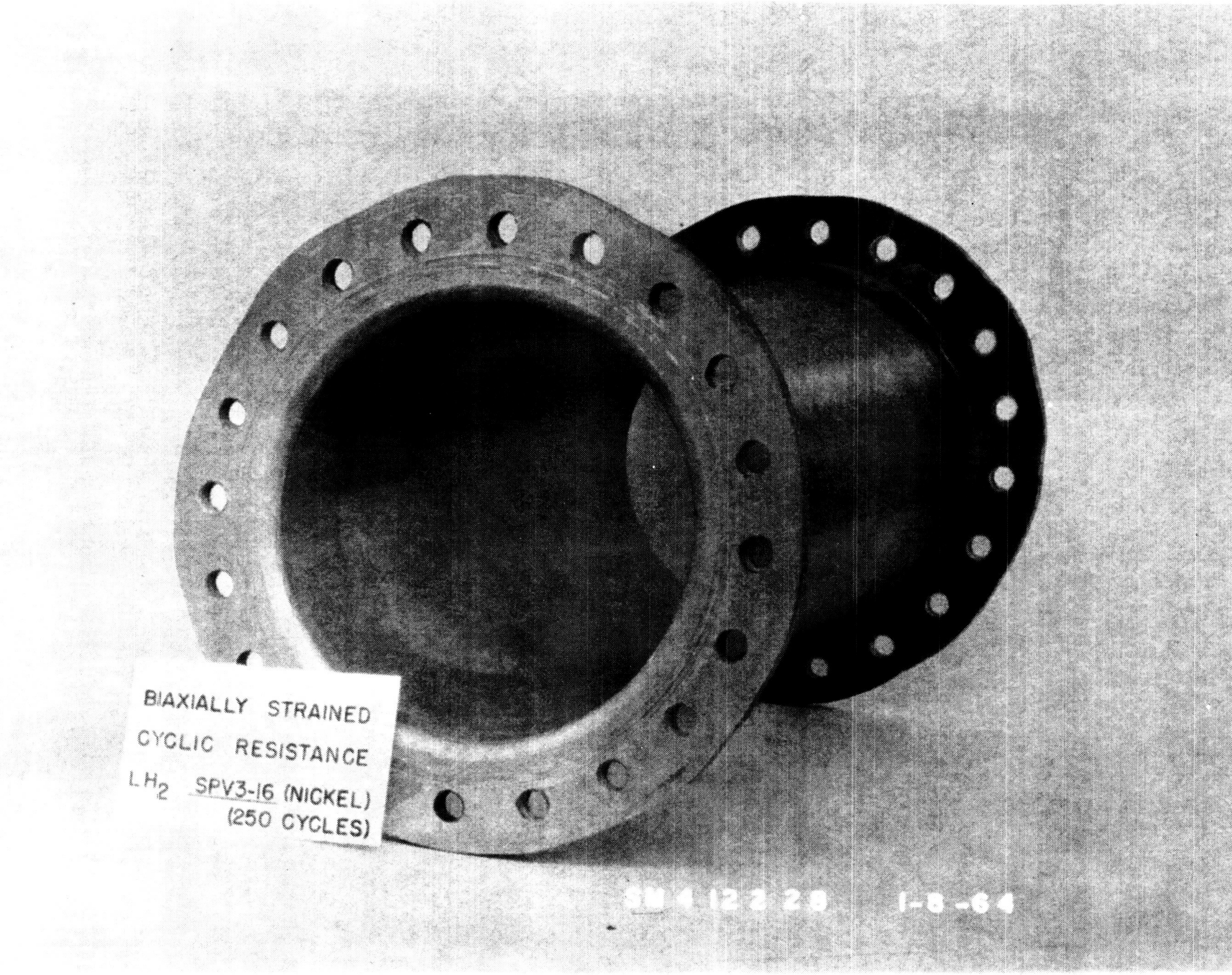
A burst test at an internal pressure of 1647 psi had been made during the second quarter with a 5 mil electrodeposited-nickel liner (SPV3-14'). Other tests with Tedlar (SPV1-13) and H-Film (SPV1-20) lined specimens showed high leakage for these materials at 164 psi and 388 psi respectively.

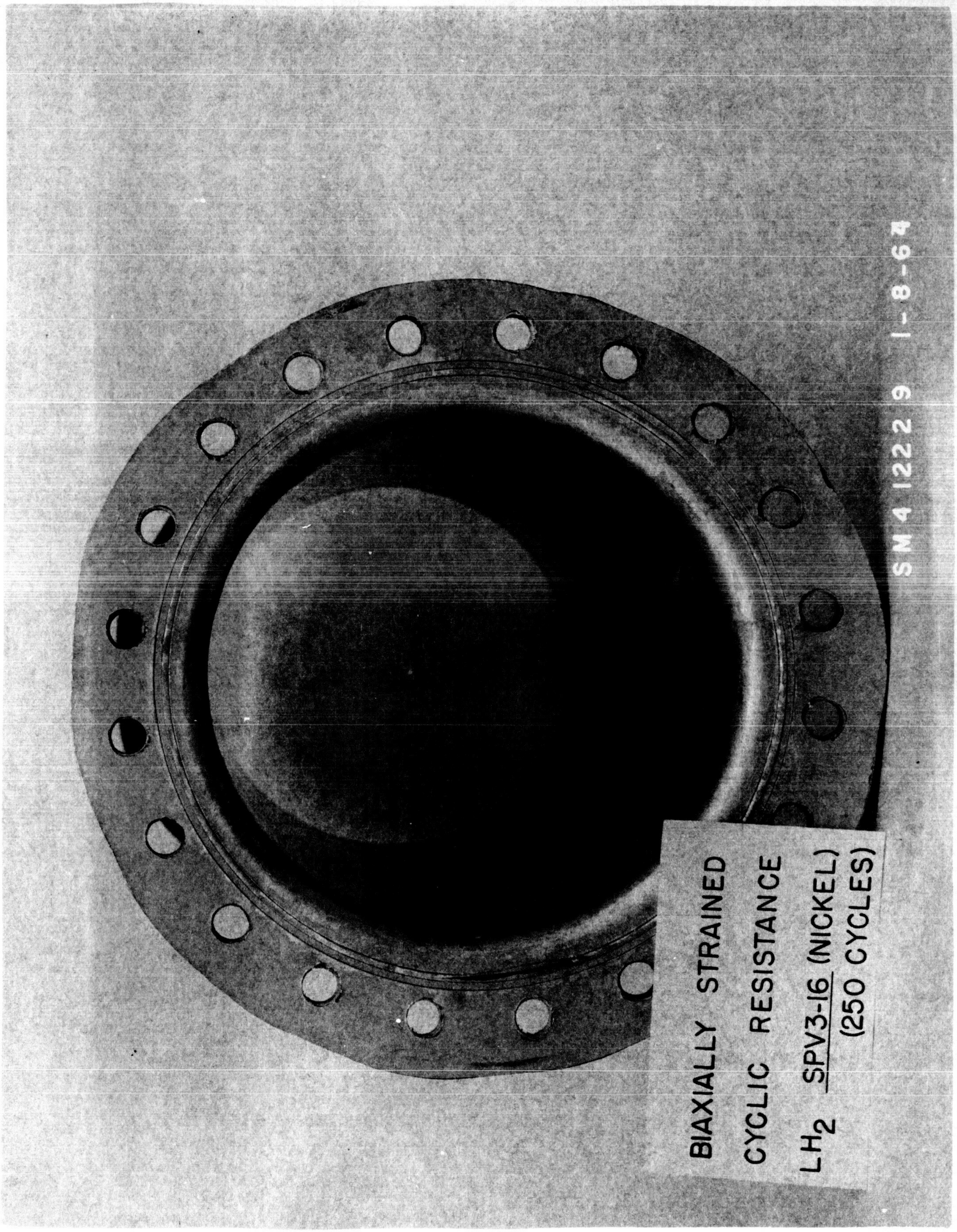
A 5 mil nickel lined specimen (SPV3-16), figure 1, was cycled 250 times at 600 spi (approximately 36% of ultimate). There was no apparent gross leakage during the test. Subsequent examination revealed neither buckles nor wrinkles; however, at one spot, cold gas appeared to have gotten between the liner and wall and upon heating to ambient temperature, "popped" a portion of the liner, figures 2 and 3. Pressurization rate corresponded to an approximate strain rate of 2.2%/min. Mechanical properties data for the specimen is given in Section 2.2.3.

A glass flake lined specimen (SPV2-9) was cycled 8 times. However, leakage was so bad that pump pressurization to the desired 600 psi was impossible and a helium gas boost was used. Respective pressurization points for each cycle are shown in figure 4. Pressurization rates of 1.0%/min and 7.61%/min respectively. Figures 5, 6, and 7 show the appearance of the liner after the test. Mechanical properties data for the specimen is given in Section 2.2.3.

A Mylar lined specimen (SPV1-17) was cycled four times. The helium boost had to be used again since the pump could not handle the excessive leakage.




BIAXIALLY STRAINED
CYCLIC RESISTANCE
LH2 SPV3-16 (NICKEL)
(250 CYCLES)
1-8-64



5 Mil Nickel Lined Specimen (SPV3-16)
After 250 cycles at 36% of Ultimate

FIGURE 3

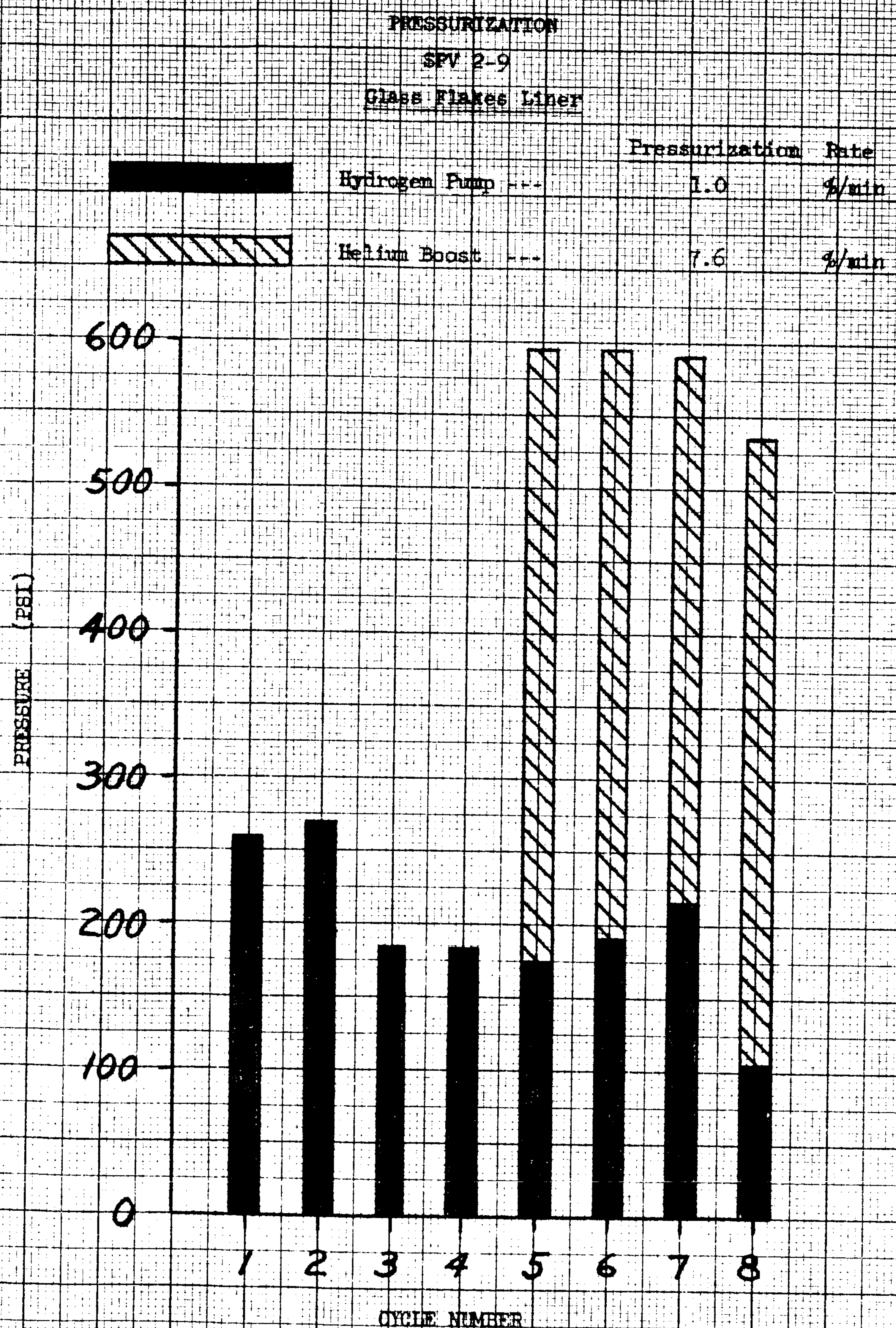
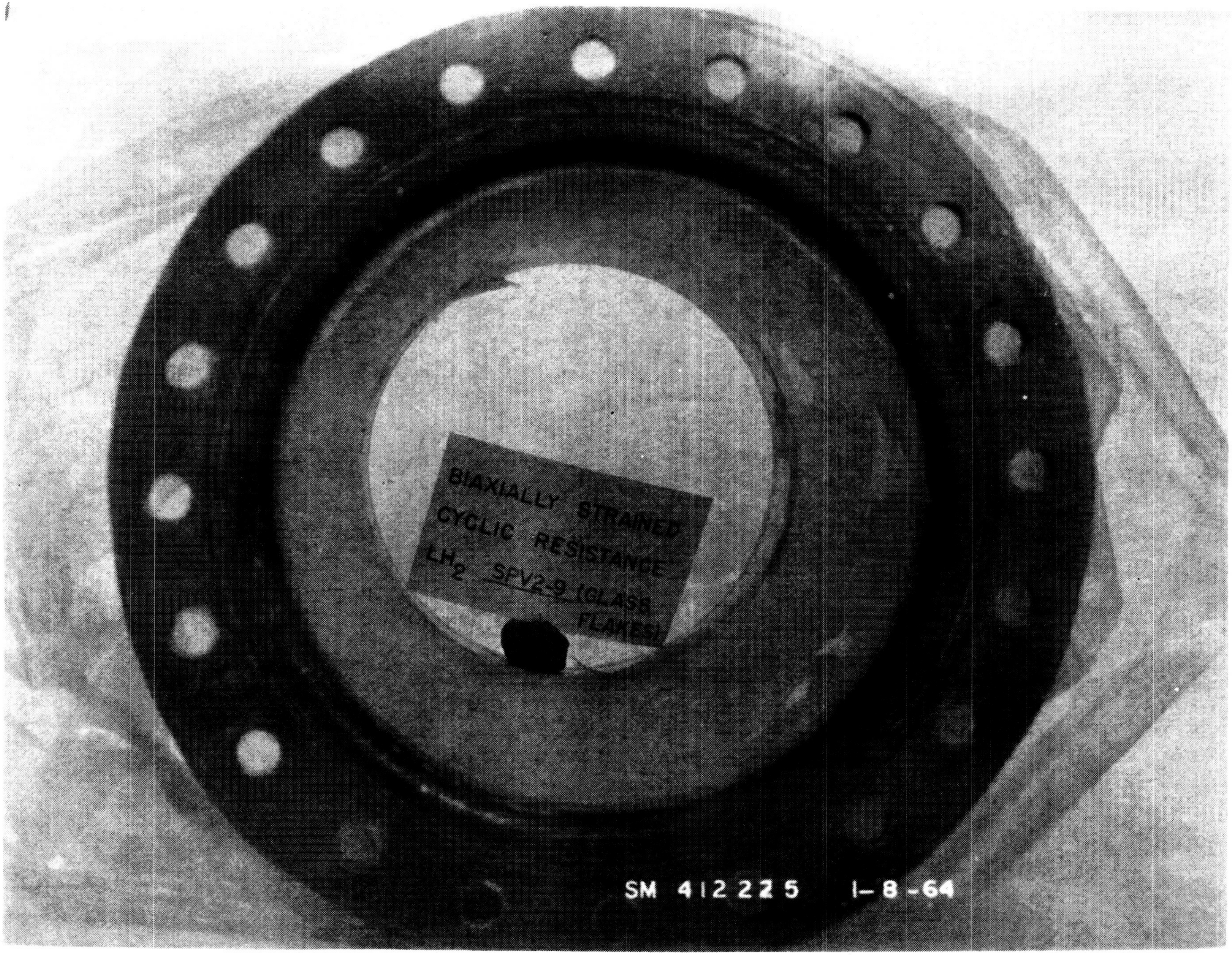
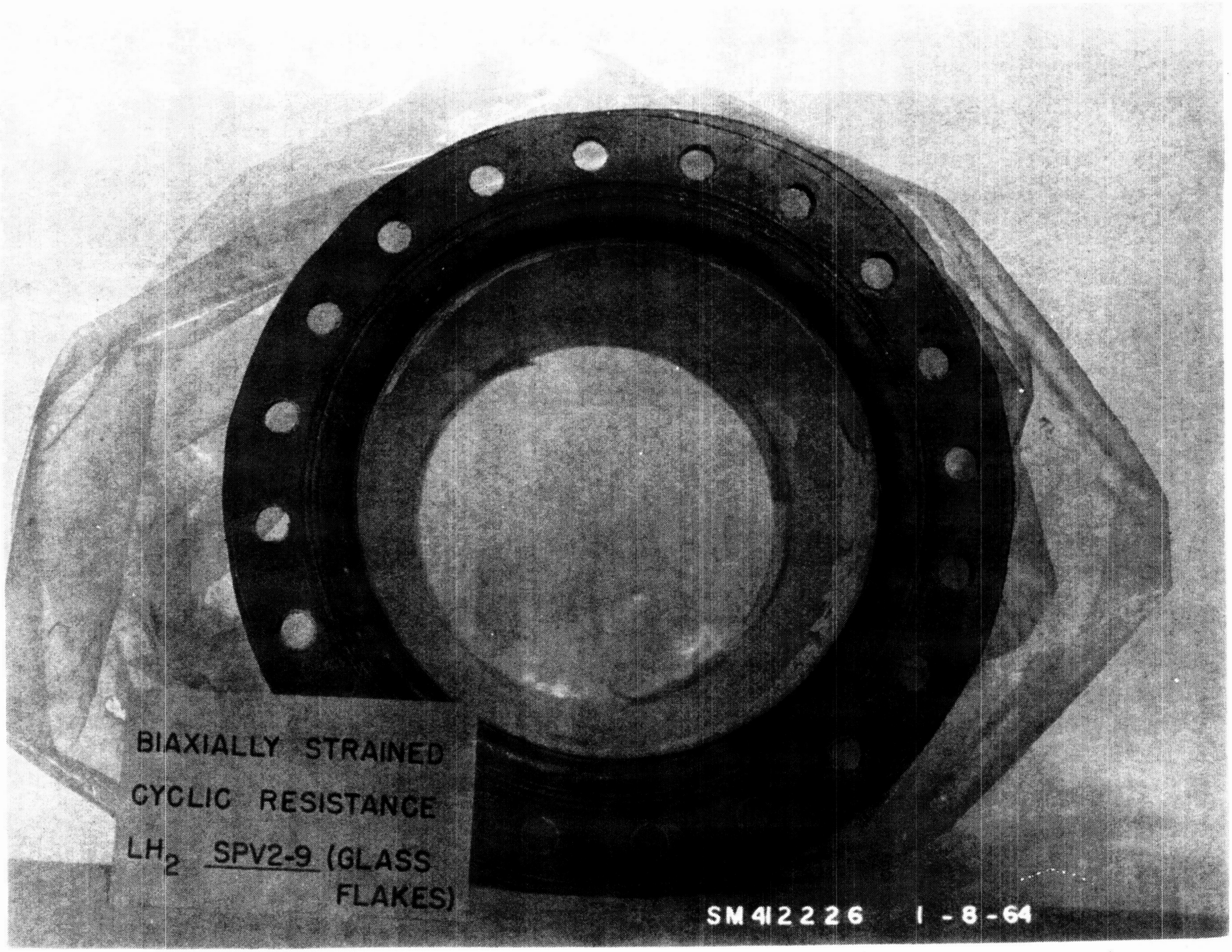

PRESSURIZATION
SFV 2-9
Glass Flakes Liner
Pressurization Rate
Hydrogen Pump --- 1.0 %/min
Helium Boost --- 7.6 %/min
600
500
400
300
200
100
0
PRESSURE (PSI)
1
2
3
4
5
6
7
8
CYCLE NUMBER

FIGURE 4


BIAXIALLY STRAINED
CYCLIC RESISTANCE
LH2 SPV2-9 (GLASS
FLAKES)
SM 412227 1-8-64


BIAXIALLY STRAINED
CYCLIC RESISTANCE
LH2 SPV2-9 (GLASS
FLAKES)
SM 412225 1-8-64


BIAXIALLY STRAINED
CYCLIC RESISTANCE
LH2 SPV2-9 (GLASS
FLAKES)
SM 412226 1-8-64

Pressurization points for each cycle are shown in figure 8. Strain rates of 2.6%/min and 4.2%/min were obtained. The liner was badly cracked in both the hoop and longitudinal directions as can be seen in figures 9 and 10.

It appears that the Mylar elongation data reported by Mowers (reference 3) was not representative of the material used for this project. Taking into consideration the contraction and childown strains, an ultimate pressure of 1000 psi and 60% cycling pressure of 600 psi would have been realized. Using the Mylar data of this project, which had been considered suspect, the predicted liner failure would have occurred at 345 psi. The liner actually leaked excessively at 360 psi. Mechanical properties data for the specimen is given in Section 2.2.3.

An H-Film lined specimen (SPV2-21) was cycled three times with a helium boost. The specimen leaked excessively at a maximum pump pressure of 355 psi. The pressurization history is shown in figure 11. Pressurization strain rates for the pump and helium were 1.1%/min and 6.9%/min respectively. The liner was badly cracked, as can be seen in figures 12 and 13. (A previously tested H-Film specimen (SPV1-20) leaked excessively at 388 psi. Barely discernible cracking was evidenced with that specimen.) Mechanical properties data for specimen SPV2-21 is given in Section 2.2.3.

An attempt was made to cycle a 5-mil nickel lined specimen (SPV3-8) at 1200 psi, which represents approximately 70% of ultimate burst pressure. Pump pressurization was inadequate to reach 1200 psi. The specimen was rechilled and the helium boost was used; the specimen went through two cycles to 1200 psi. The specimen failed at the peak of the second such cycle. It was discovered later that the wrong calibration factor had been applied to the instrument used to monitor the pressure and the last two cycles were to 1540 psi rather than the desired 1200 psi. The pressurization history is shown in figure 14 and the failed specimen in figure 15. Pressurization strain rates for the pump and helium were 3.3%/min and 19.1%/min respectively. Mechanical properties data is given in Section 2.2.3.

Tables 1 and 2 summarize the testing to date of the biaxial vessels and liner behavior. As can be seen from Table 2, no polymer has been capable of

PRESSURIZATION

SPV 1-17

Mylar "A" Liner

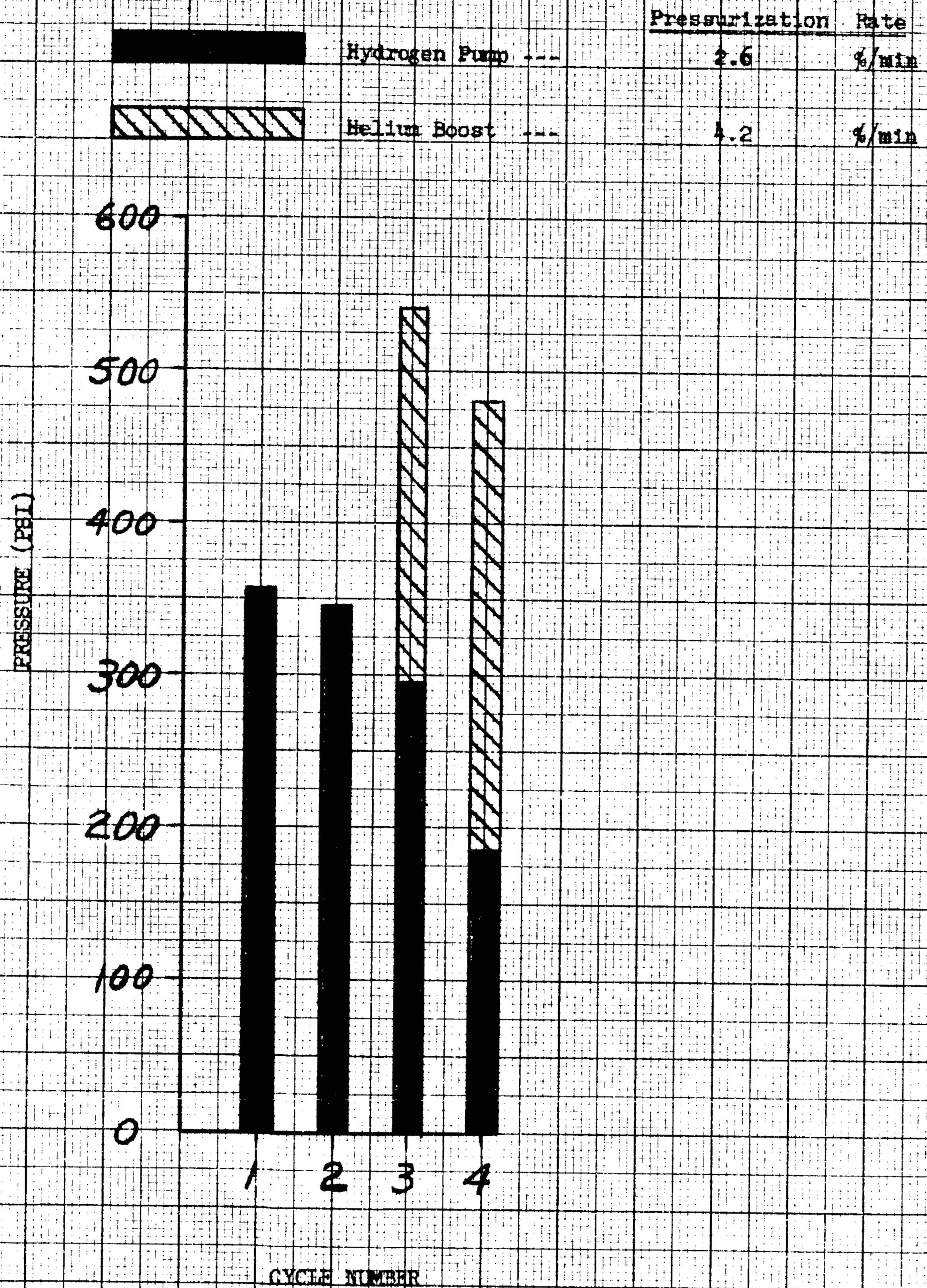
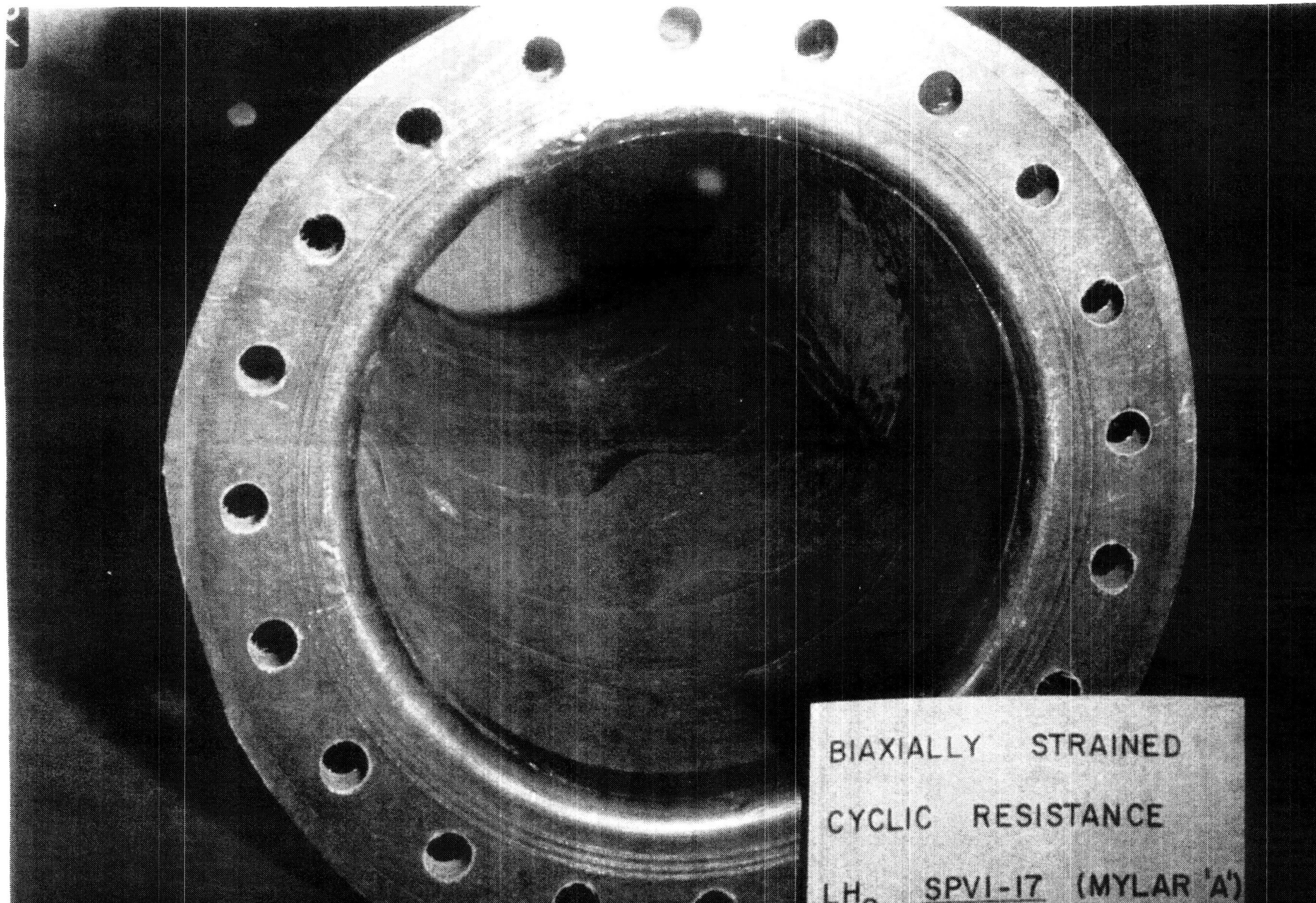
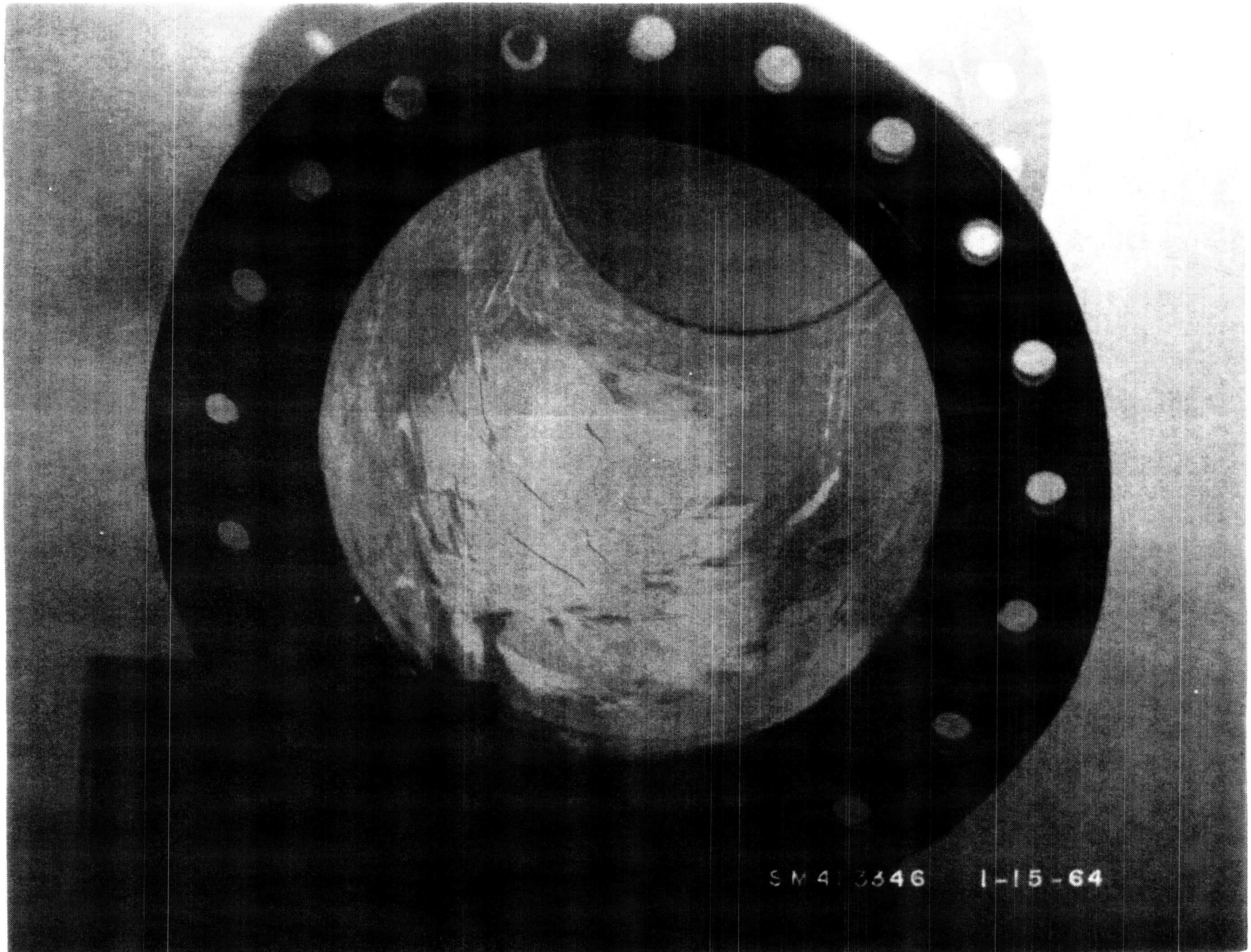


FIGURE 8

K+E 10 X 10 TO THE 1/2 INCH 359T-11 KEUFFEL & ESSER CO.


BIAXIALLY STRAINED
CYCLIC RESISTANCE
LH2 SPVI-17 (MYLAR 'A')
SM 413345 1-15-64


SM41 3346 1-15-64

PRESSURIZATION

SPV 2-21

H-Film Liner

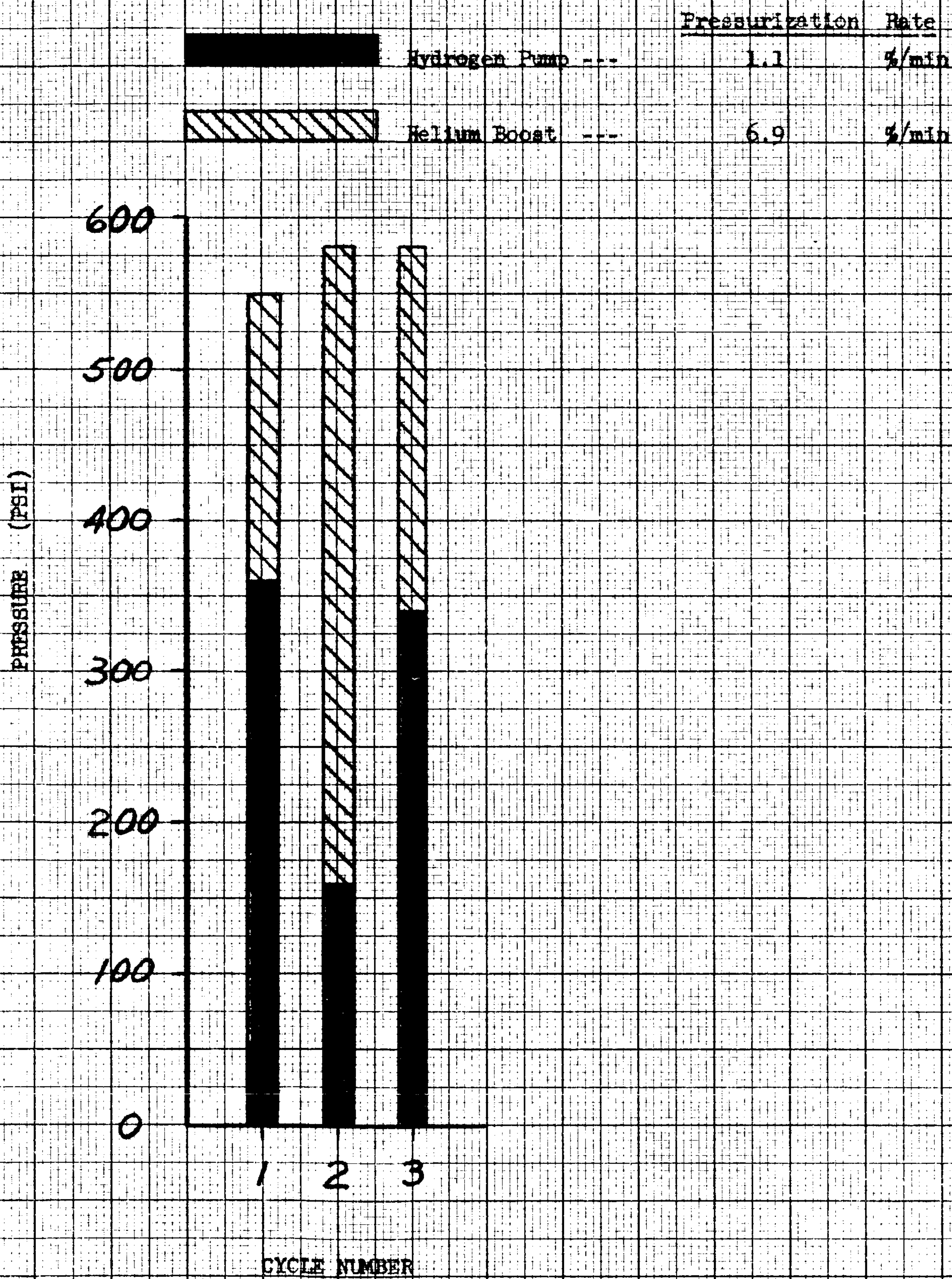
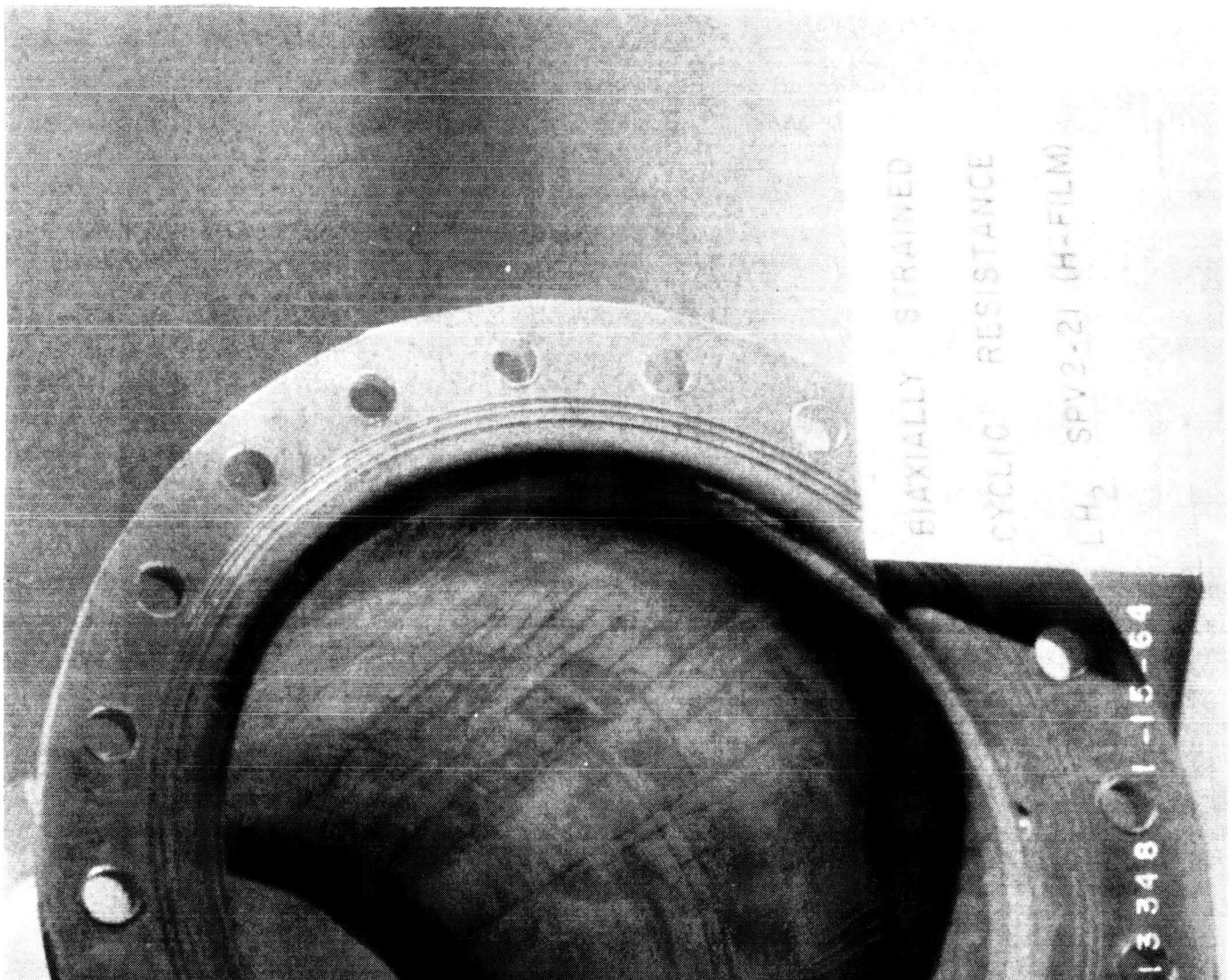
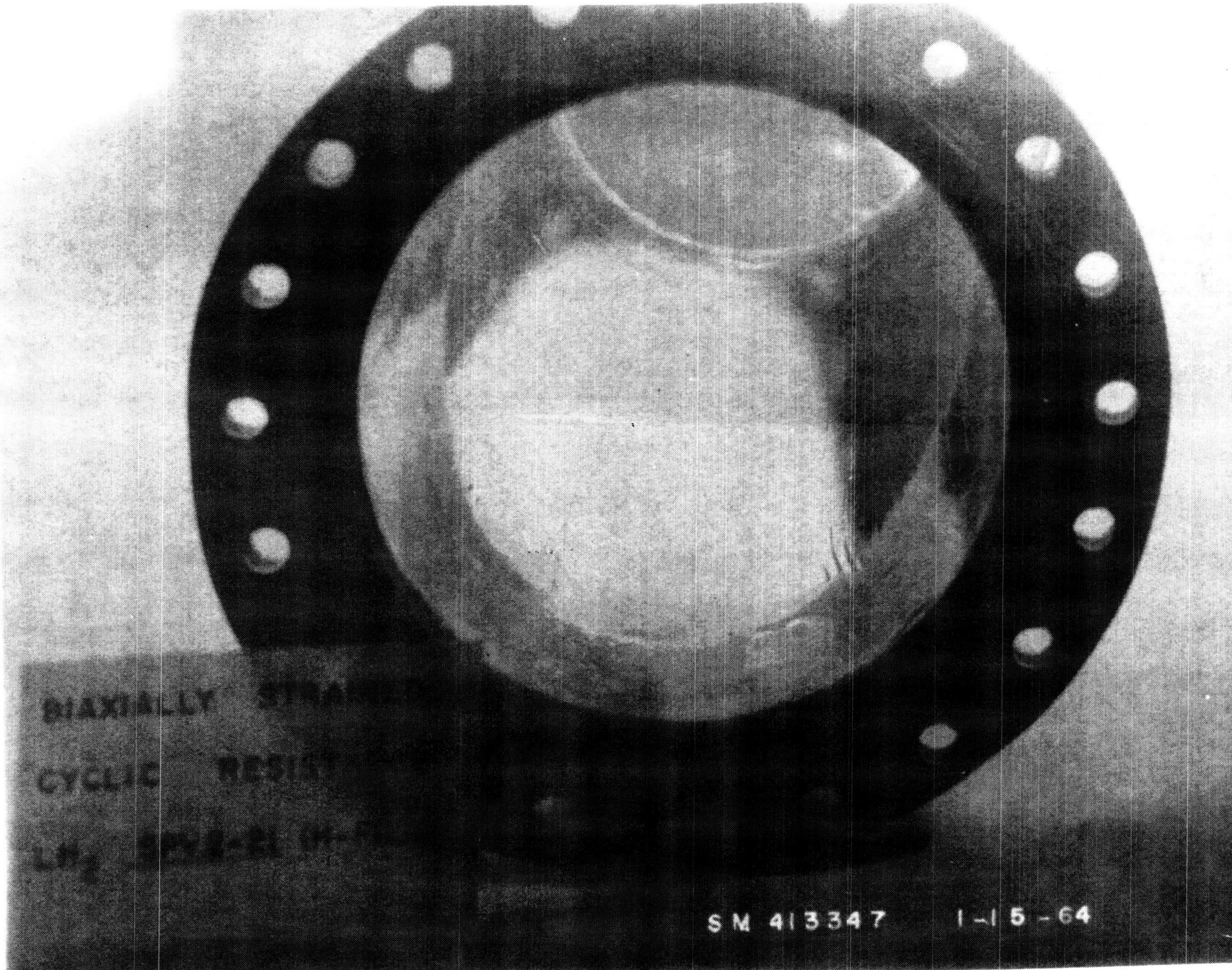


FIGURE 11

K+E 10 X 10 TO THE 1/2 INCH 359-11 KEUFFEL & ESSER CO.


BIAXIALLY STRAINED
CYCLIC RESISTANCE
LH2 SPV2-21 (H-FILM)
SM 413348 1-15-64


BIAXIALLY
CYCLIC
SM 413347 1-15-64

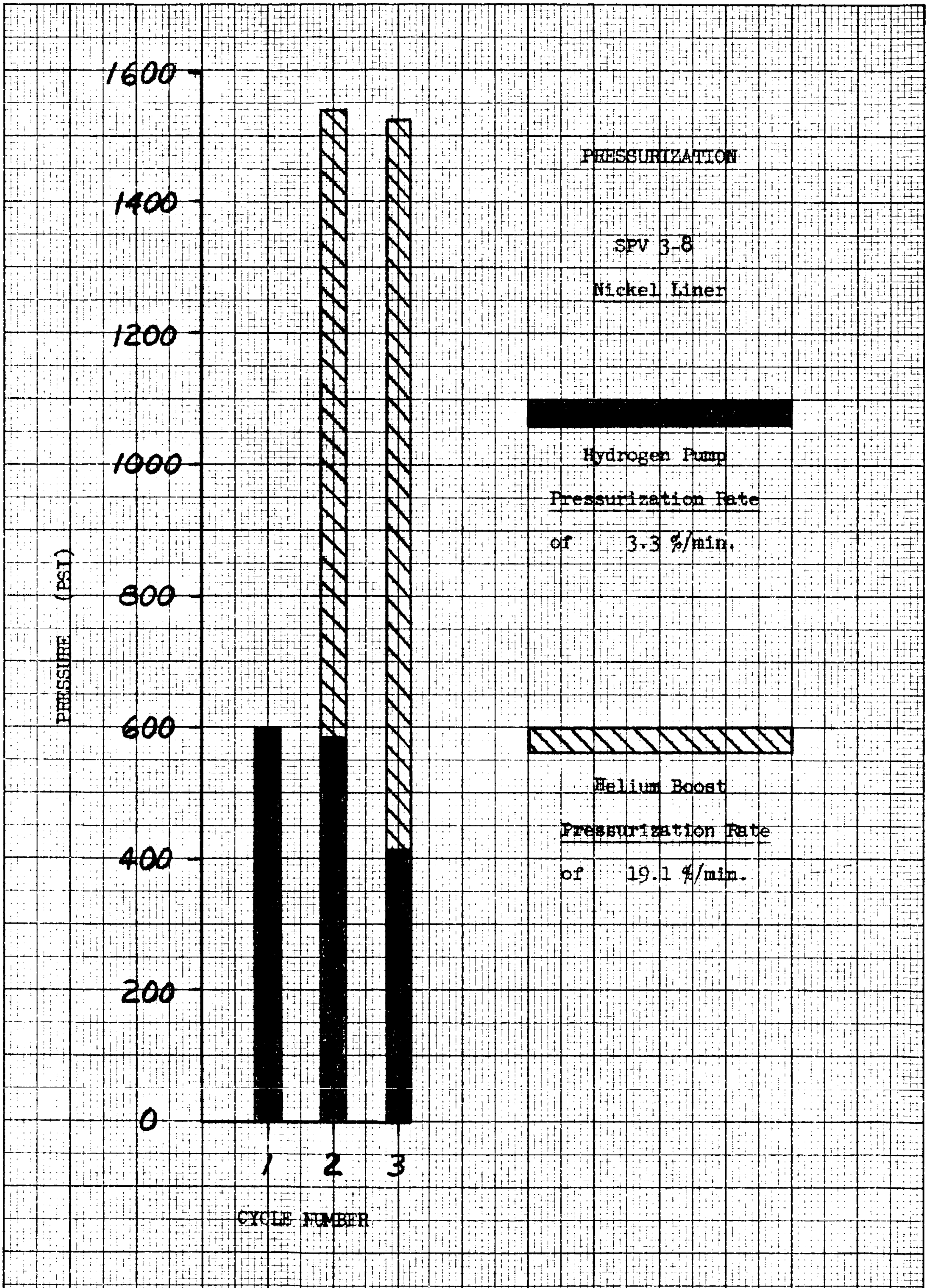


FIGURE 14

K+E 10 X 10 TO THE ½ INCH 359T-11 KEUFFEL & ESSER CO.


NAS 3-2562
BIAXIALLY STRAINED
CYCLIC RESISTANCE
LH2 SPV3-8 (NICKEL)
SM413349 1-15-64

-15

TABLE 1

7 1/2-INCH DIAMETER TESTING

(TO DATE 3-31-64)

LINER	SPECIMEN NO.	TEMP (^{o}F)	TEST	AIM (psi)	MAXIMUM ACTUAL (psi)	RESULT	TOTAL CYCLES
Nickel	SPV1-11	70	Burst	N.A.*	748	Leakage	4
Polurethane	SPV1-3	70	Burst	N.A.	N.A.	Uninstrumented burst	1
Polyurethane	SPV1-10	70	Burst	N.A.	1100	Burst	1
Mylar "A"	SPV2-1'	70	Permeability	660	353	Leakage	3
Tedlar	SPV1-5	70	Permeability	660	660	Good test	21
Nickel	SPV3-4	70	Pearmeability	660	450	Leakage	3
Nickel	SPV3-14'	-423	Burst	N.A.	1647	Good test-burst	20
Tedlar	SPV1-13	-423	Burst	N.A.	184	Leakage	1
H-Film	SPV1-20	-423	Permeability and Cyclic	1000	388	Leakage	4
Silver	SPV1-24	-423	Cyclic	600	600	Testing not completed	76
Nickel	SPV3-16	-423	Cyclic	600	600	Good test	250
Glass Flakes	SPV2-9	-423	Cyclic	600	270 600	Pump He boost	8
Mylar "A"	SPV1-17	-423	Cyclic	600	360 540	Pump He boost	4
H-Film	SPV2-21	-423	Cyclic	600	355 585	Pump He boost	3
Nickel	SPV3-8	-423	Cyclic	1200	611 1540	Pump He boost	2
Silver	SPV2-2	-423	Cyclic	1200	249 695	Pump He boost	4

*Not available at time of test

TABLE 2

	TEDLAR	H-FILM	GLASS FLAKES	MYLAR "A"	POLY-URETHANE
Contraction (10^{-3} in/in)	9.16	5.26	2.35	3.86	16.31
Childown (%)	0.73	0.34	0.04	0.20	1.44
Average Ultimate Elongation (%)	2.16	1.69	2.70	0.82	2.20
Residual Elongation (%)	1.43	1.35	2.66	0.62	0.76
Predicted Liner Failure (psi)	786	742	1461	345	418
% of Cylinder Ultimate	48	45	89	21	25
Actual Liner Failure (psi)	184	355-388	270	360	——
% of Cylinder Ultimate	11	21-24	16	22	——

sustaining a load higher than 25% of the ultimate strength of the biaxial cylinder. Table 3 shows the status of the 7 1/2-inch diameter biaxial specimens fabricated as of March 31, 1964.

On the basis of the results to date, metallic liners appear to show the most promise. Due to its excellent cyclic behavior at 36% of ultimate, most favorable differential contraction compatibility, and most favorable deposition characteristics of the three metallic candidates, electrodeposited nickel will be used as the liner material for the 18-inch diameter pressure vessels.

2.2 FIBER GLASS/RESIN COMPOSITE INVESTIGATION

Three resin systems were selected for evaluation during the course of the project: ERLA 0510/ZZLO803, EPI-REZ 510-5042/EPI CURE 841, and EPI-REZ 5101/APCO 322.

Preliminary results (reference 1) indicated that ERLA 0510/ZZLO803 would be most suitable for the initial biaxial liner screening tests. All of the 7 1/2-inch diameter biaxial specimens have been fabricated of this resin system and S994 glass fibers. Based upon the preliminary data and experience in handling this resin system, ERLA 0510/ZZLO803 has been chosen, with the advice and approval of the NASA/Lewis Program Manager, for use in fabricating the 18-inch diameter pressure vessels.

It was reported from a recent filament winding conference that the HTS finish caused a degradation of the glass if not fully protected from environmental conditions. Since all of the glass in this project utilizes HTS finish and much of the material was on hand, a quality control check was made to ascertain any deterioration of the stored material. Comparison of six glass spools revealed no apparent degradation of the material between the original quality control check in September, and the recent check in February. All of the material for this project is stored in a well sealed box, which contains a desiccant.

2.2.2 Uniaxial Cyclic Tests at Cryogenic Temperatures

This work was completed during the first quarter (reference 1).

TABLE 3

7 1/2-INCH DIAMETER TEST SPECIMEN STATUS*

SPECIMEN NO.	LINER	STATUS
SPV1-1	Mylar	Damaged-available
2-1'	Mylar	Tested
2-2	Silver	Tested
1-3	Polyurethane	Tested uninstrumented burst
3-4	Nickel	Tested
1-5	Tedlar	Tested
2-6	Copper	Available
1-7	H-Film	Available
3-8	Nickel	Tested
2-9	Glass Flakes	Tested
3-10	Polyurethane	Tested
1-11	Nickel	Tested
3-12	Tedlar	Available
1-13	Tedlar	Tested
2-14	Nickel	Ruined
3-14'	Nickel	Tested
1-15	Mylar	Available
3-16	Nickel	Tested
1-17	Mylar	Tested
1-18	Polyurethane	Available
2-19	Copper	Available
1-20	H-Film	Tested
2-21	H-Film	Tested
2-22	Polyurethane	Available
2-23	Polyurethane	Available
1-24	Silver	Tested

*Fabrication as of 3-31-64

2.2.3 Mechanical Properties and Cyclic Tests

Mechanical properties tests are being made of the selected materials using the 7 1/2-inch diameter biaxial test specimen. This specimen permits complete evaluation of the resin/fiber glass system under conditions of actual use in the vessels; i.e., resin strains and cracks in two directions.

All tests are performed in the vacuum test chamber. Measurements are made of pressure, hoop elongation, and longitudinal elongation. Hoop and longitudinal elongations for the nickel lined specimen SPV3-16 (250 cycles) are shown in figures 16 and 17. As can be seen, a set is introduced into the structural wall and the modulus increases with increasing cycles. No attempt has been made to reduce the data to a stress-strain diagram due to the complications of determining the liner strength, which probably has changed and either become higher (strain hardening) or lower (Bauschinger strain softening) due to the prestraining and recycling into the plastic regions. An examination of pertinent literature (references 4 through 10) has revealed the complexity of predicting the structural behavior of the material; addition of the cryogenic environment creates further complications. Smith, et al (reference 11) report that "(low and intermediate life range) cyclic stress-strain relations have been established and are - - - - substantially different from the virgin tensile data - - - -. - - - - fair correlation was obtained between the degree of cyclic strain hardening and softening (at ambient temperature) and the ratio of ultimate strength over (0.2%) yield strength. Hardening always took place when (the) ratio exceeded 1.4 and softening occurred when the ratio was less than 1.2." The uniaxial tensile data for electrodeposited nickel (reference 1) at -423°F gives the ultimate strength/0.2% yield strength as 1.22.

Stress-strain diagrams for the glass-flakes lined specimen (SFV2-9), Mylar "A" lined specimen (SFV1-17), and H-Film lined specimen (SFV2-21) are shown in figures 18 though 35. Liner strengths have been subtracted on the basis of loading from an unstrained condition for each cycle. Hoop elongations for the nickel lined specimen SPV3-8 is shown in figure 36. The two cycles at 93% of ultimate agree with the predictions, which were based on ambient temperature data, of Young (reference 12), and Wolff and Siuta (reference 13), and Patterson (reference 14), figure 37.

BIAXIAL TEST
PRESSURE VS ELONGATION
(SPECIMEN SPV3-16)

LIQUID HYDROGEN (-423°F)
CYCLES 1, 50, 100, 150, 199 & 250

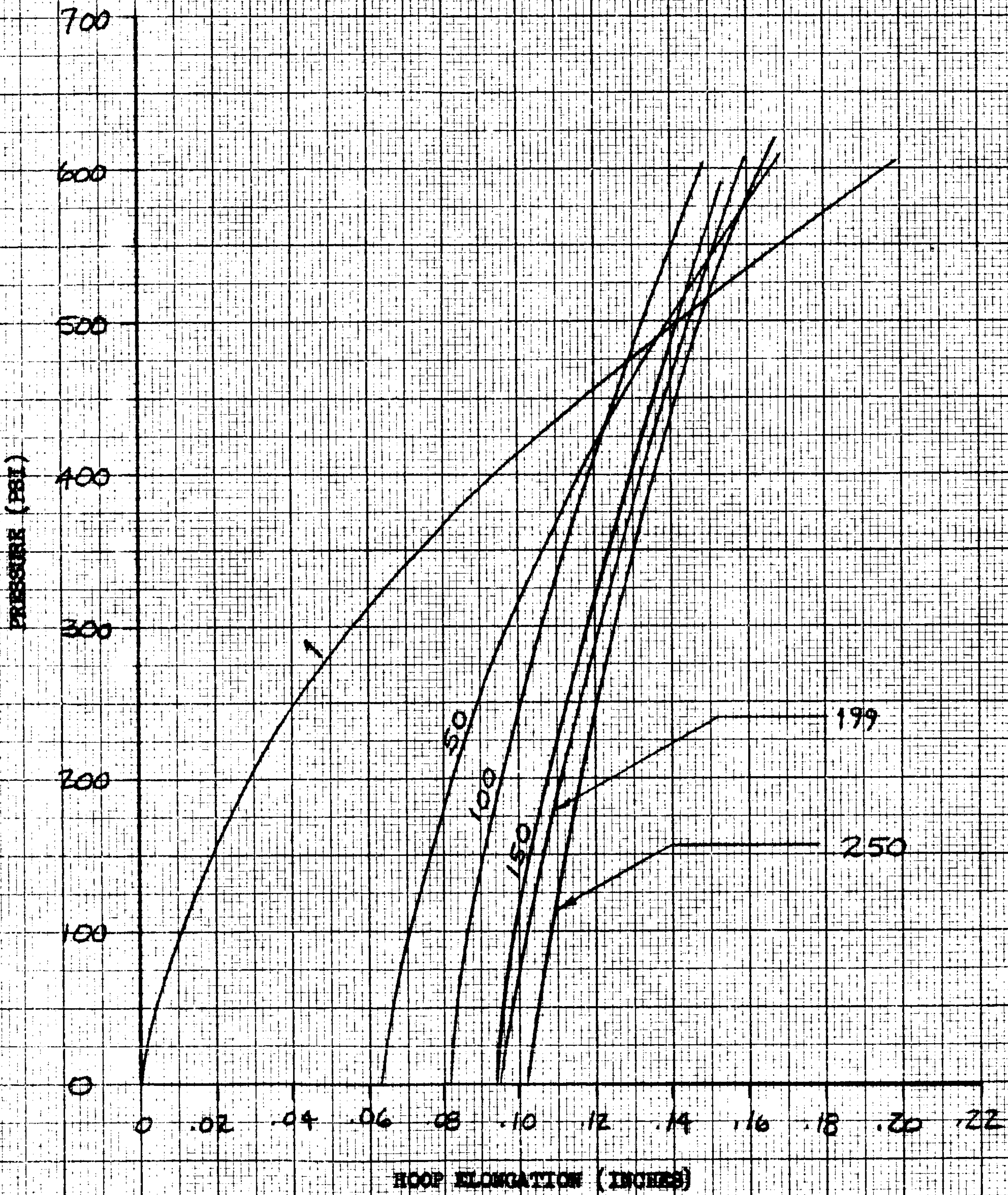


FIGURE 16

K+E 10 X 10 TO THE 1/2 INCH 359T-11
KEUFFEL & ESSER CO. ALBANENE® MADE IN U.S.A.

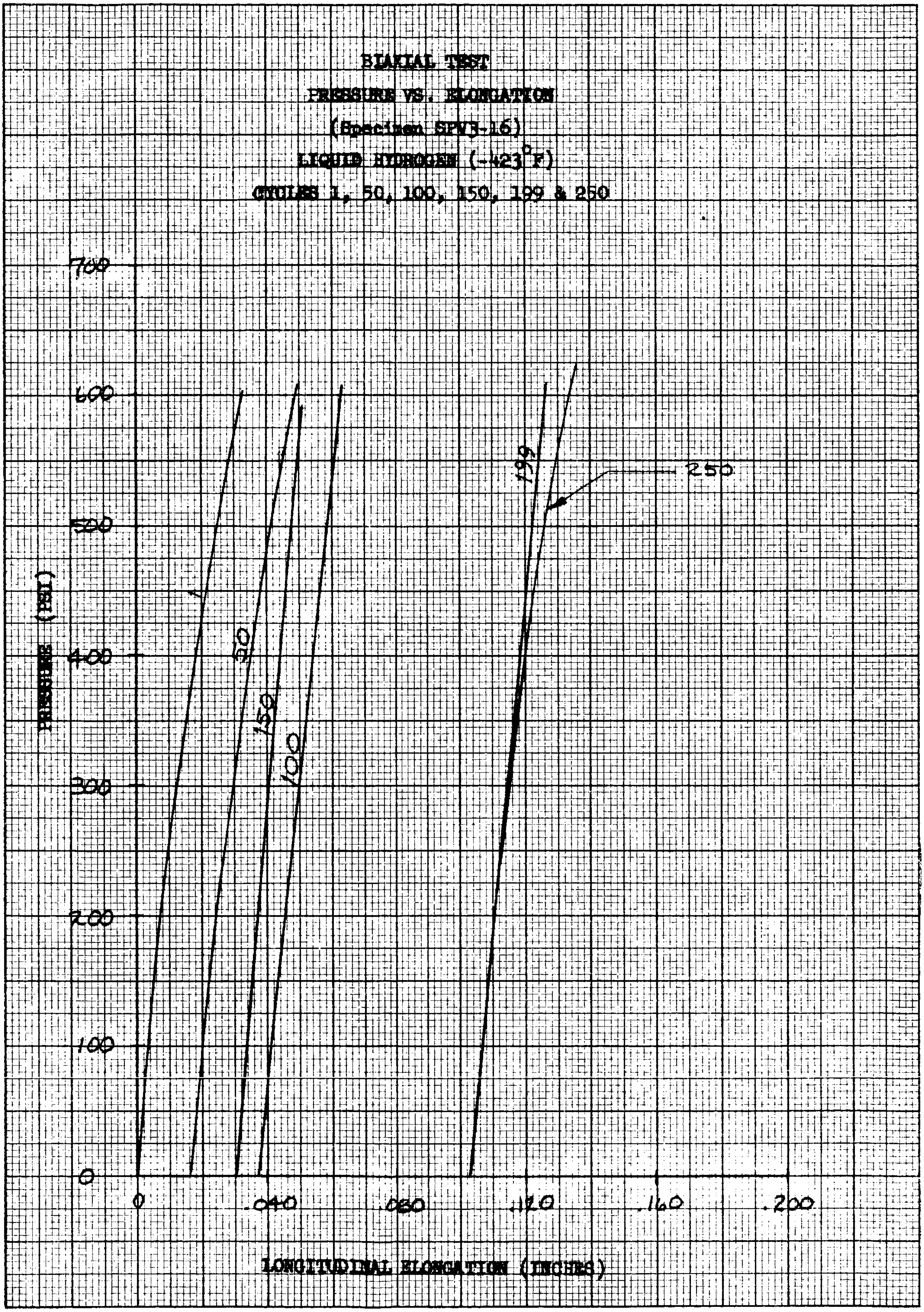

BIAXIAL TEST
PRESSURE VS. ELONGATION
(Specimen SPVB-16)
LIQUID HYDROGEN (-423°F)
CYCLES 1, 50, 100, 150, 199 & 250
700
600
500
400
300
200
100
0
PRESSURE (PSI)
1
50
150
100
199
250
0
.040
.080
.120
.160
.200
LONGITUDINAL ELONGATION (INCHES)

FIGURE 17

BIAXIAL TEST
STRESS-STRAIN DIAGRAM
GLASS FLAKES LINER (Specimen SPV2-9)
LIQUID HYDROGEN (-423°F)
INITIAL CYCLE
PRESSURE 249 PSI

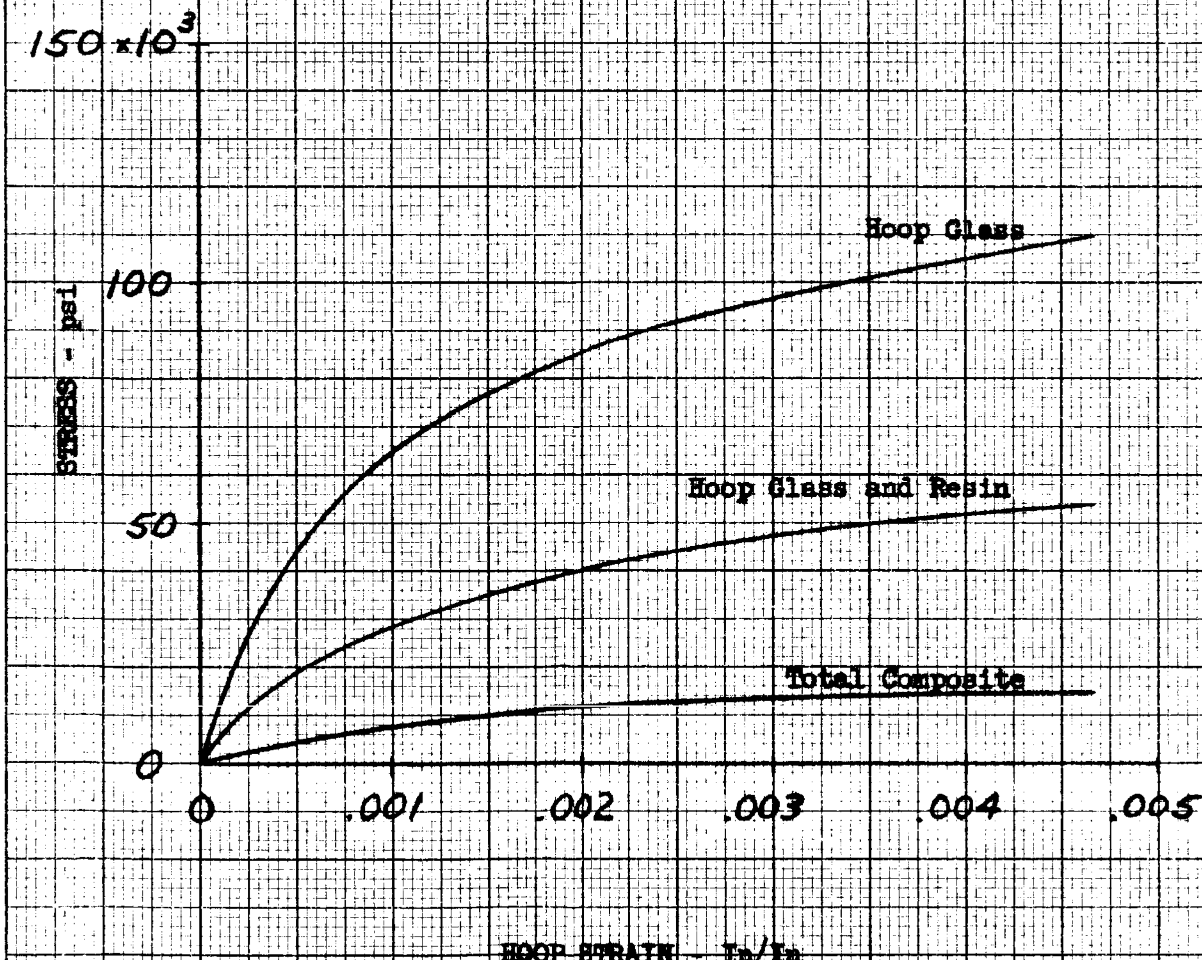


FIGURE 18

K+E 10 X 10 TO THE 1/2 INCH 359T-11 KEUFFEL & ESSER CO.

BIAXIAL TEST
STRESS-STRAIN DIAGRAM
GLASS FLAKES LINER (Specimen SPV2-9)
LIQUID HYDROGEN (-423 F)
INITIAL CYCLE
PRESSURE 249 PSI

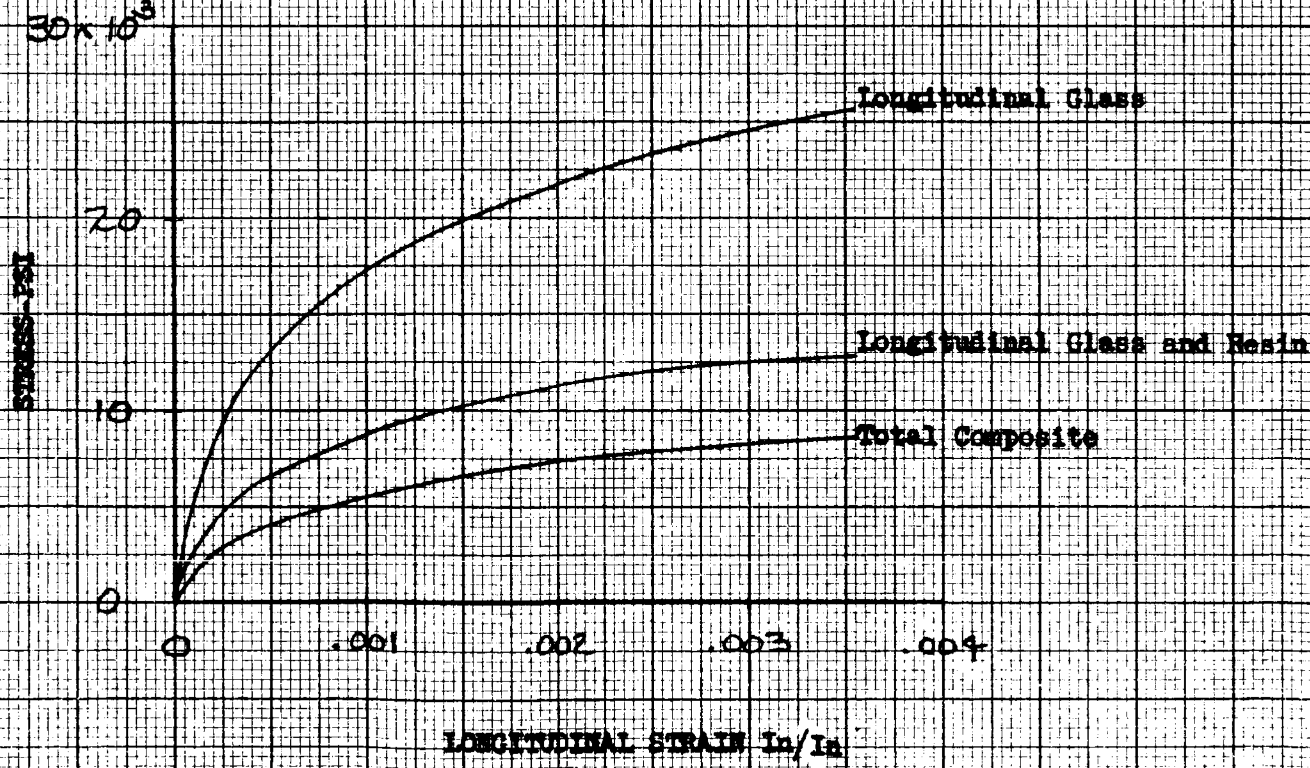
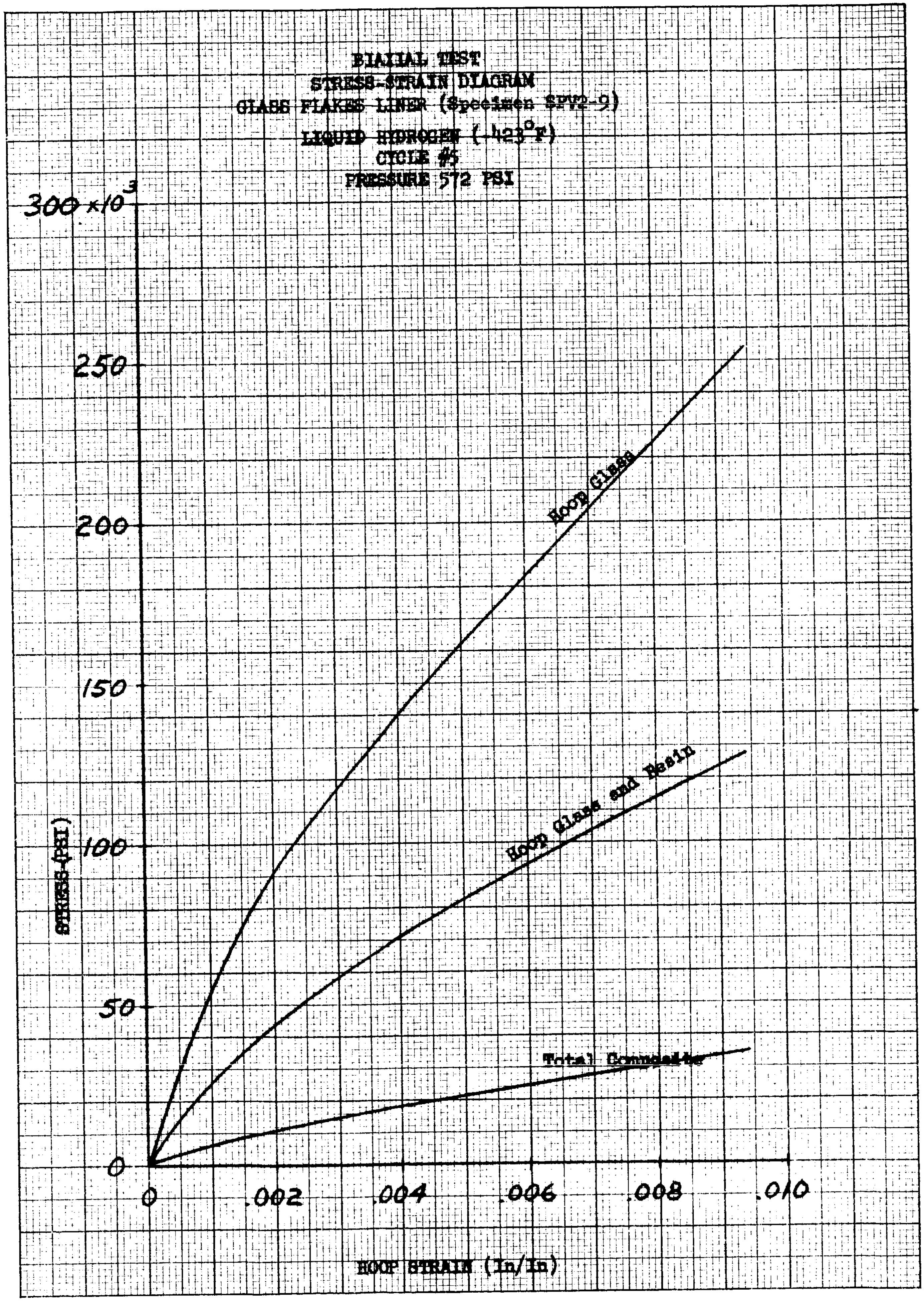
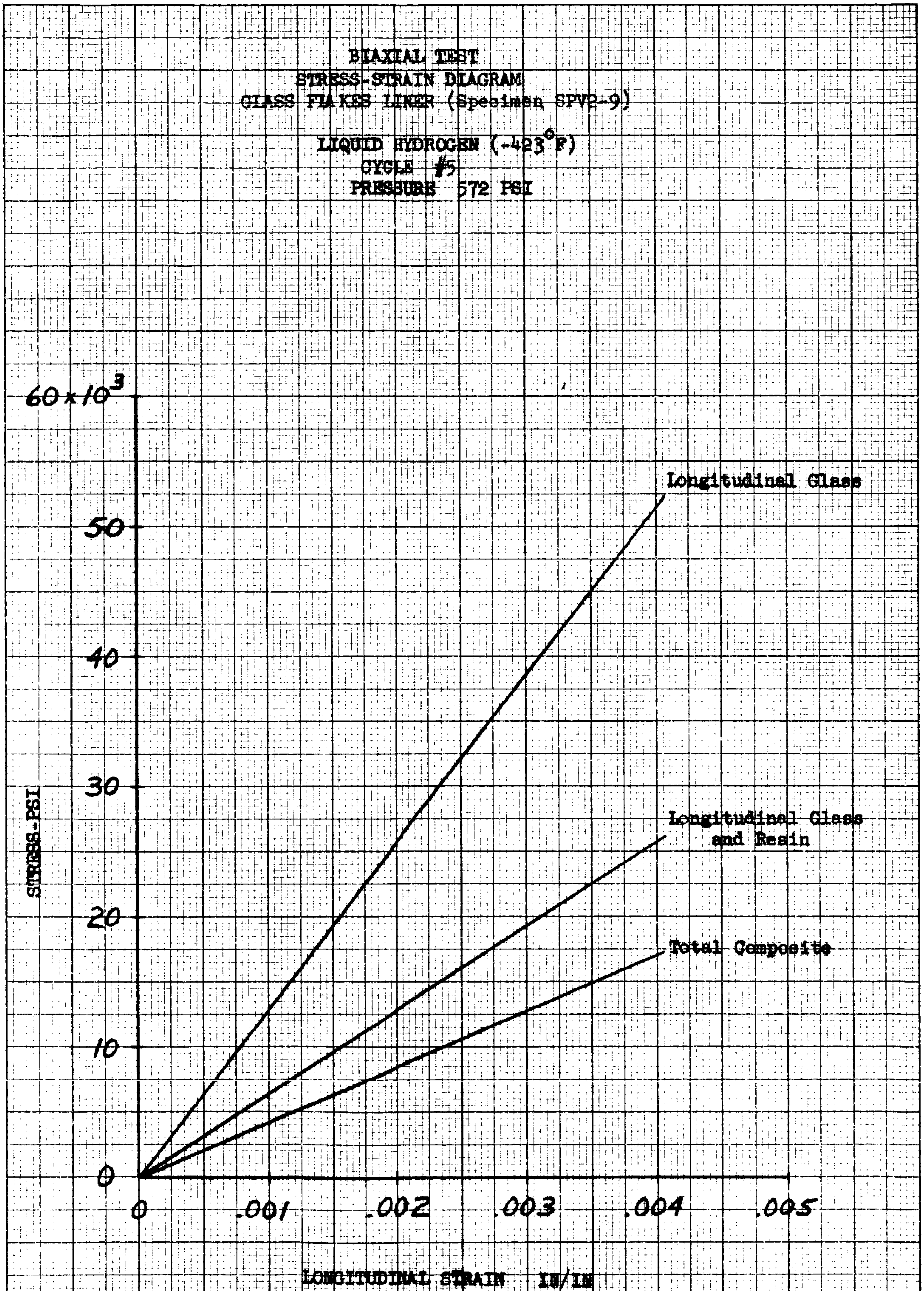


FIGURE 19



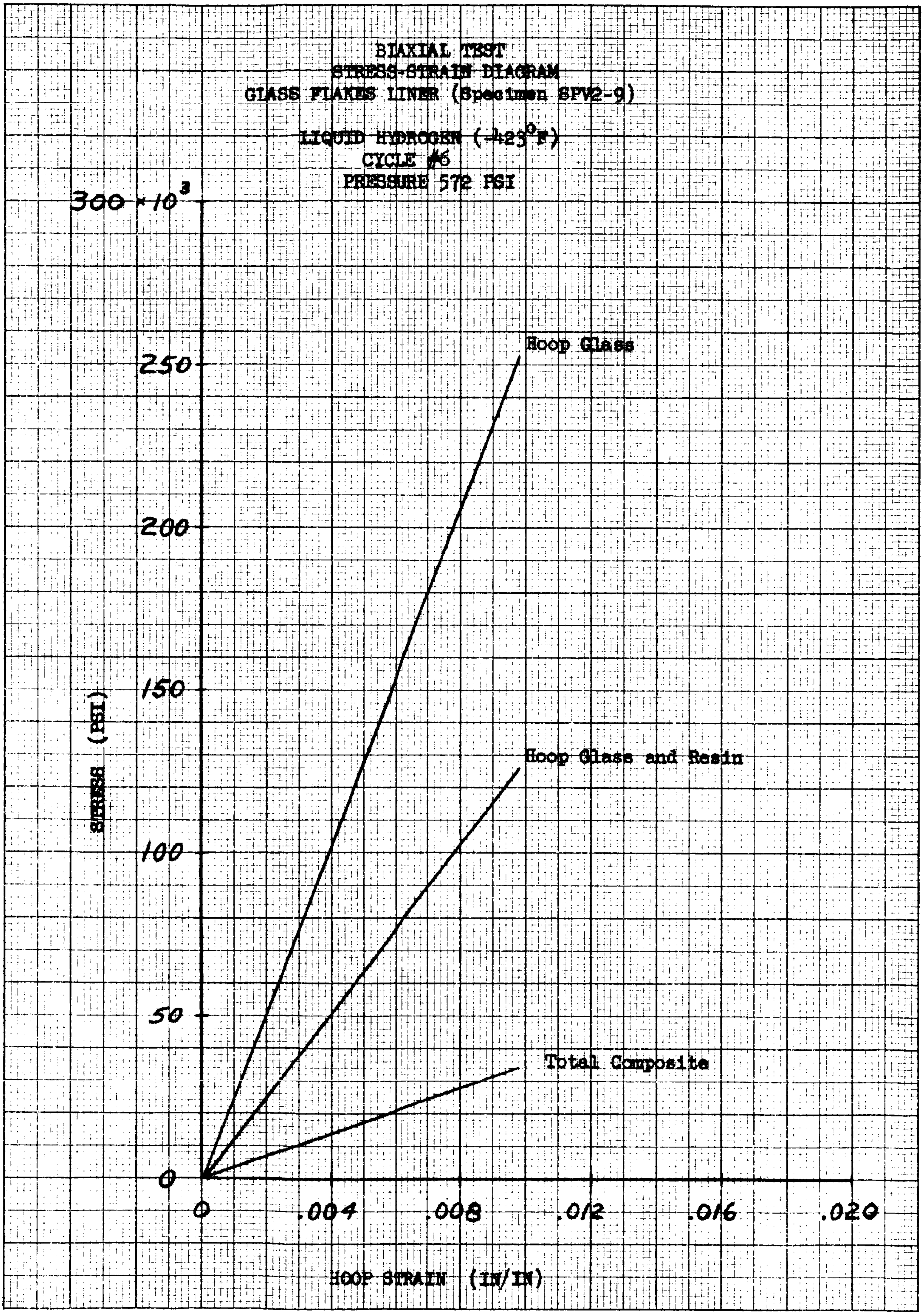
BIAXIAL TEST
STRESS-STRAIN DIAGRAM
GLASS FLAKES LINER (Specimen SPV2-9)
LIQUID HYDROGEN (-423°F)
CYCLE #5
PRESSURE 572 PSI
STRESS (PSI)
300 ×10^3
250
200
150
100
50
0
Hoop Glass
Hoop Glass and Resin
Total Composite
0
.002
.004
.006
.008
.010
HOOP STRAIN (In/In)

FIGURE 20



BIAXIAL TEST
STRESS-STRAIN DIAGRAM
GLASS FLAKES LINER (Specimen SPV2-9)
LIQUID HYDROGEN (-423°F)
CYCLE #5
PRESSURE 572 PSI
60 x 10^3
50
40
30
20
10
0
STRESS-PSI
Longitudinal Glass
Longitudinal Glass and Resin
Total Composite
0
.001
.002
.003
.004
.005
LONGITUDINAL STRAIN IN/IN

FIGURE 21



BIAXIAL TEST
STRESS-STRAIN DIAGRAM
GLASS FLAKES LINER (Specimen SPV2-9)
LIQUID HYDROGEN (-423°F)
CYCLE #6
PRESSURE 572 PSI
300 × 10³
250
200
150
100
50
0
STRESS (PSI)
Hoop Glass
Hoop Glass and Resin
Total Composite
0
.004
.008
.012
.016
.020
HOOP STRAIN (IN/IN)

FIGURE 22

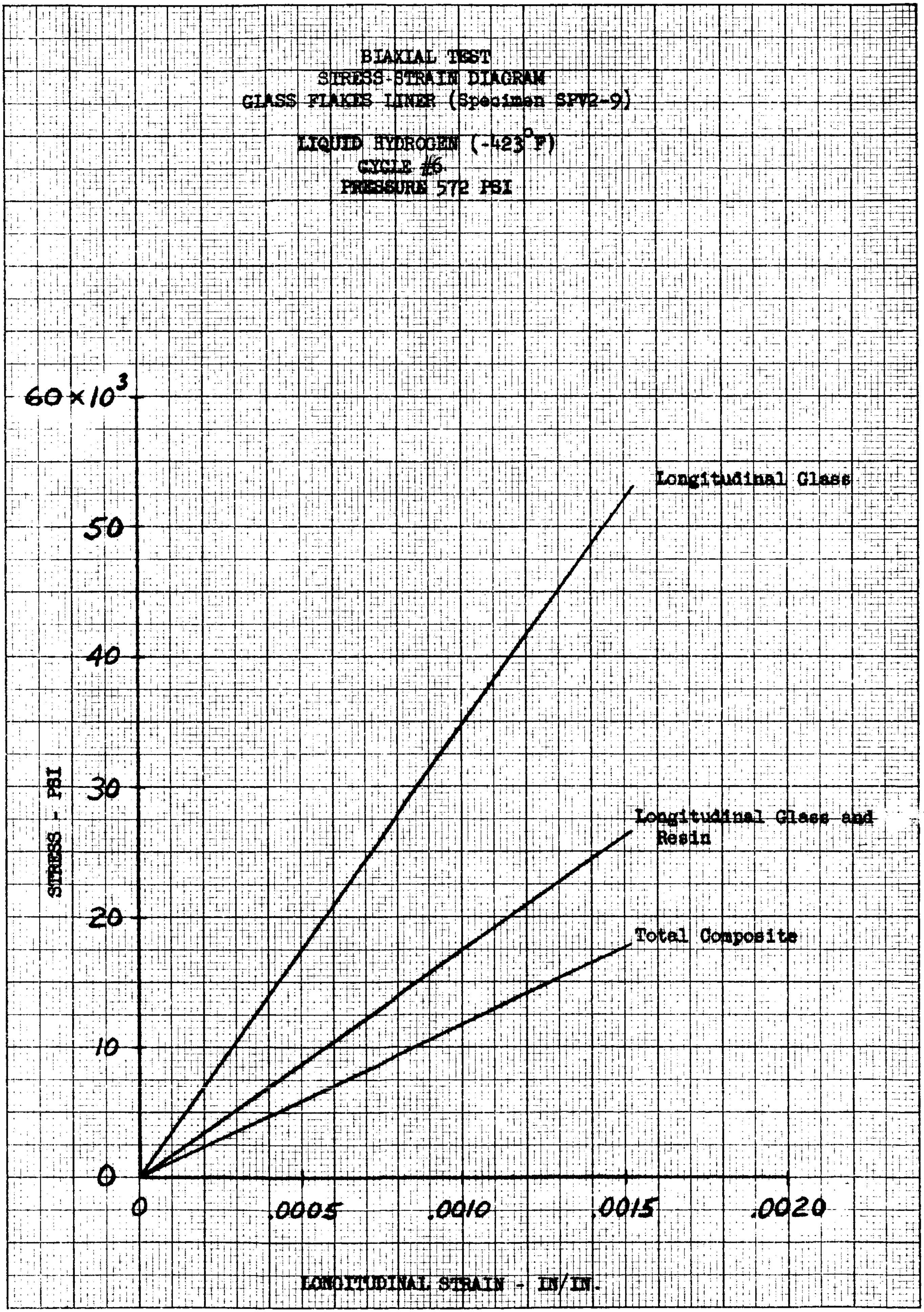

BIAXIAL TEST
STRESS-STRAIN DIAGRAM
GLASS FLAKES LINER (Specimen SPV2-9)
LIQUID HYDROGEN (-423°F)
CYCLE #6
PRESSURE 572 PSI
60×10³
50
40
30
20
10
0
STRESS - PSI
Longitudinal Glass
Longitudinal Glass and Resin
Total Composite
0
.0005
.0010
.0015
.0020
LONGITUDINAL STRAIN - IN/IN.

FIGURE 23

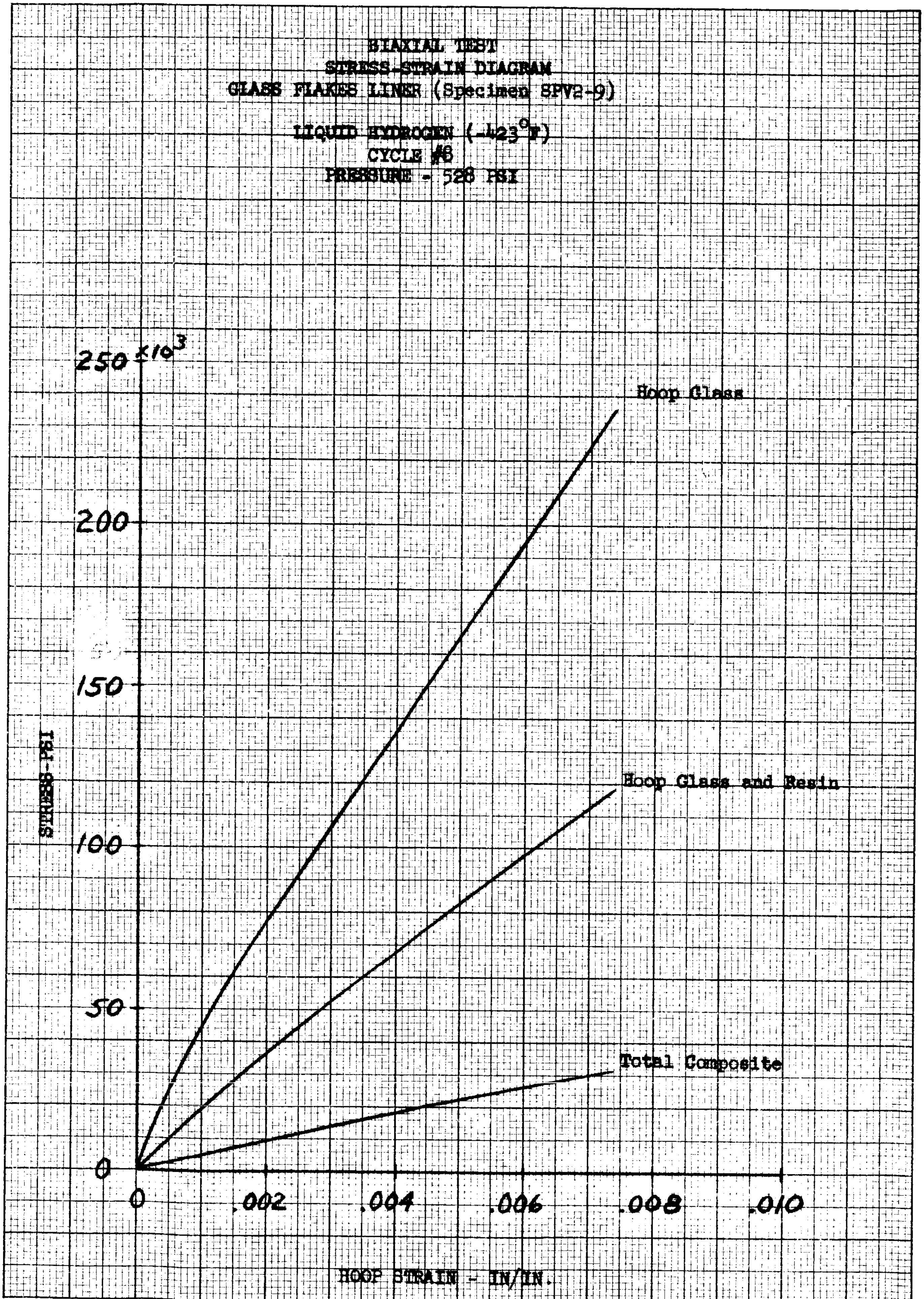

BIAXIAL TEST
STRESS-STRAIN DIAGRAM
GLASS FLAKES LINER (Specimen SFV2-9)
LIQUID HYDROGEN (-423°F)
CYCLE #8
PRESSURE - 528 PSI
250 x10^3
200
150
100
50
0
STRESS-PSI
Hoop Glass
Hoop Glass and Resin
Total Composite
0
.002
.004
.006
.008
.010
HOOP STRAIN - IN/IN.

FIGURE 24

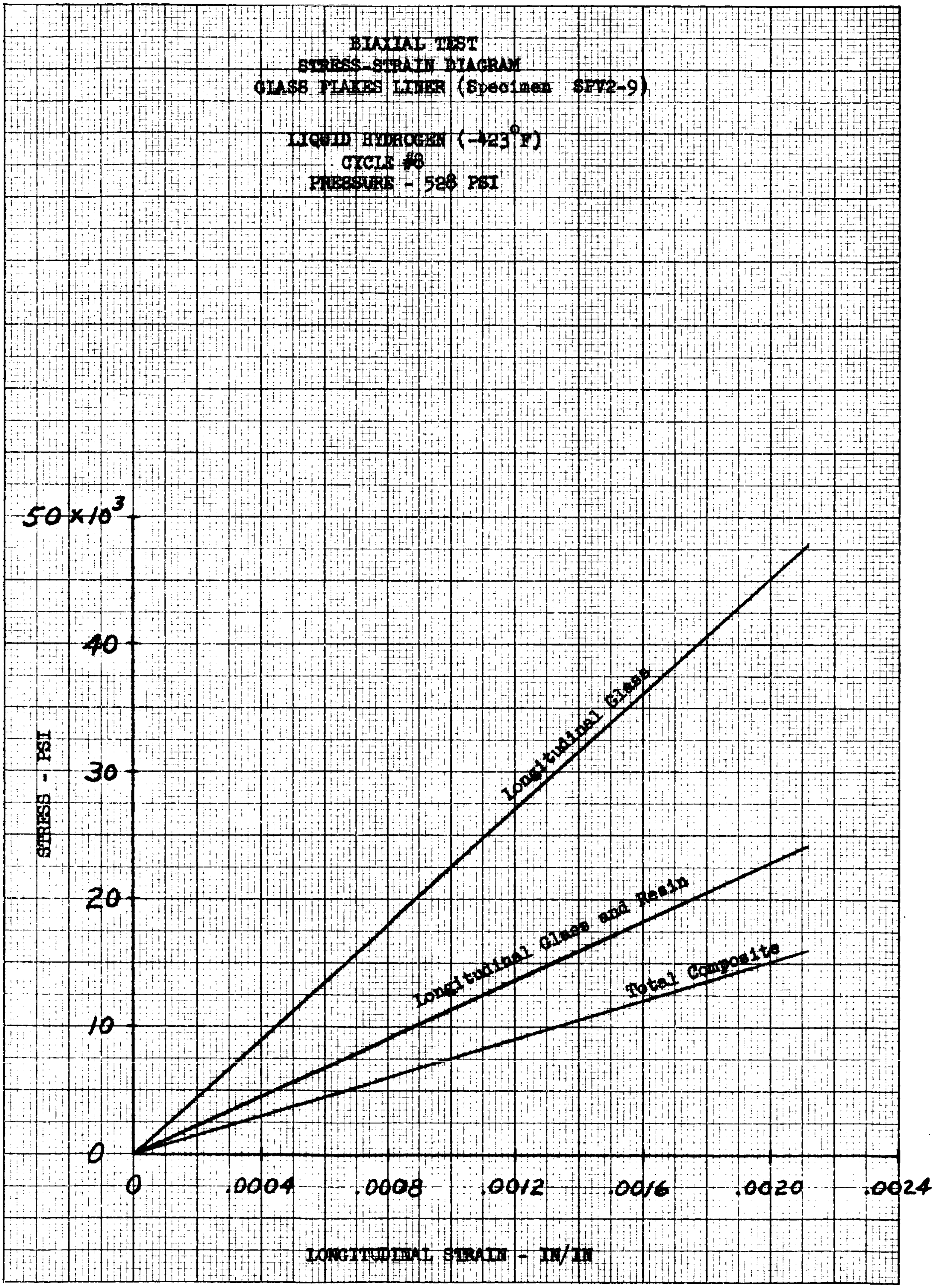


FIGURE 25

KEUFFEL & ESSER CO. 10 X 10 TO THE ½ INCH 359T-11

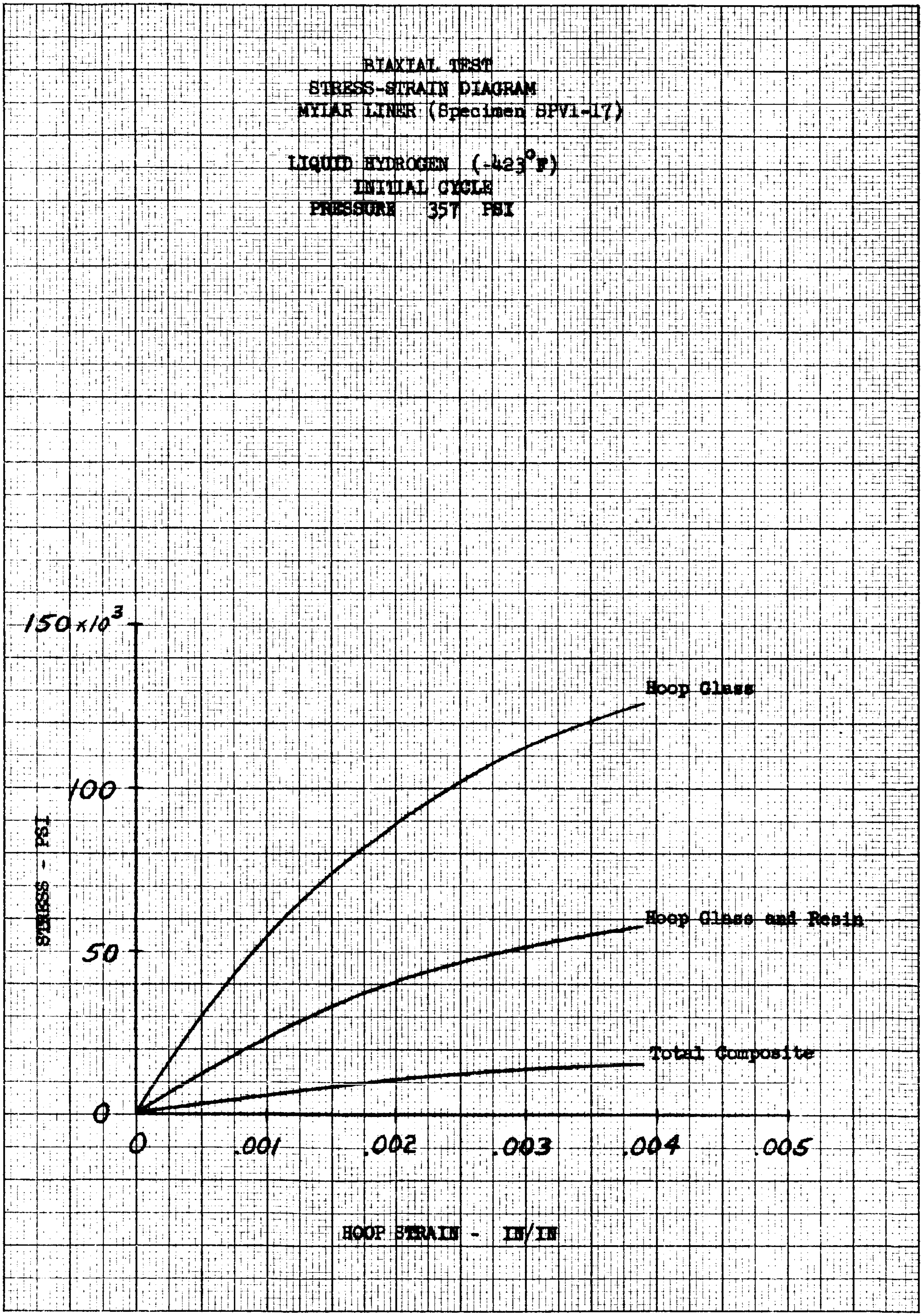

BIAXIAL TEST
STRESS-STRAIN DIAGRAM
MYLAR LINER (Specimen SPV1-17)
LIQUID HYDROGEN (-423°F)
INITIAL CYCLE
PRESSURE 357 PSI
150 x 10³
100
50
0
STRESS - PSI
Hoop Glass
Hoop Glass and Resin
Total Composite
0
.001
.002
.003
.004
.005
HOOP STRAIN - IN/IN

FIGURE 26

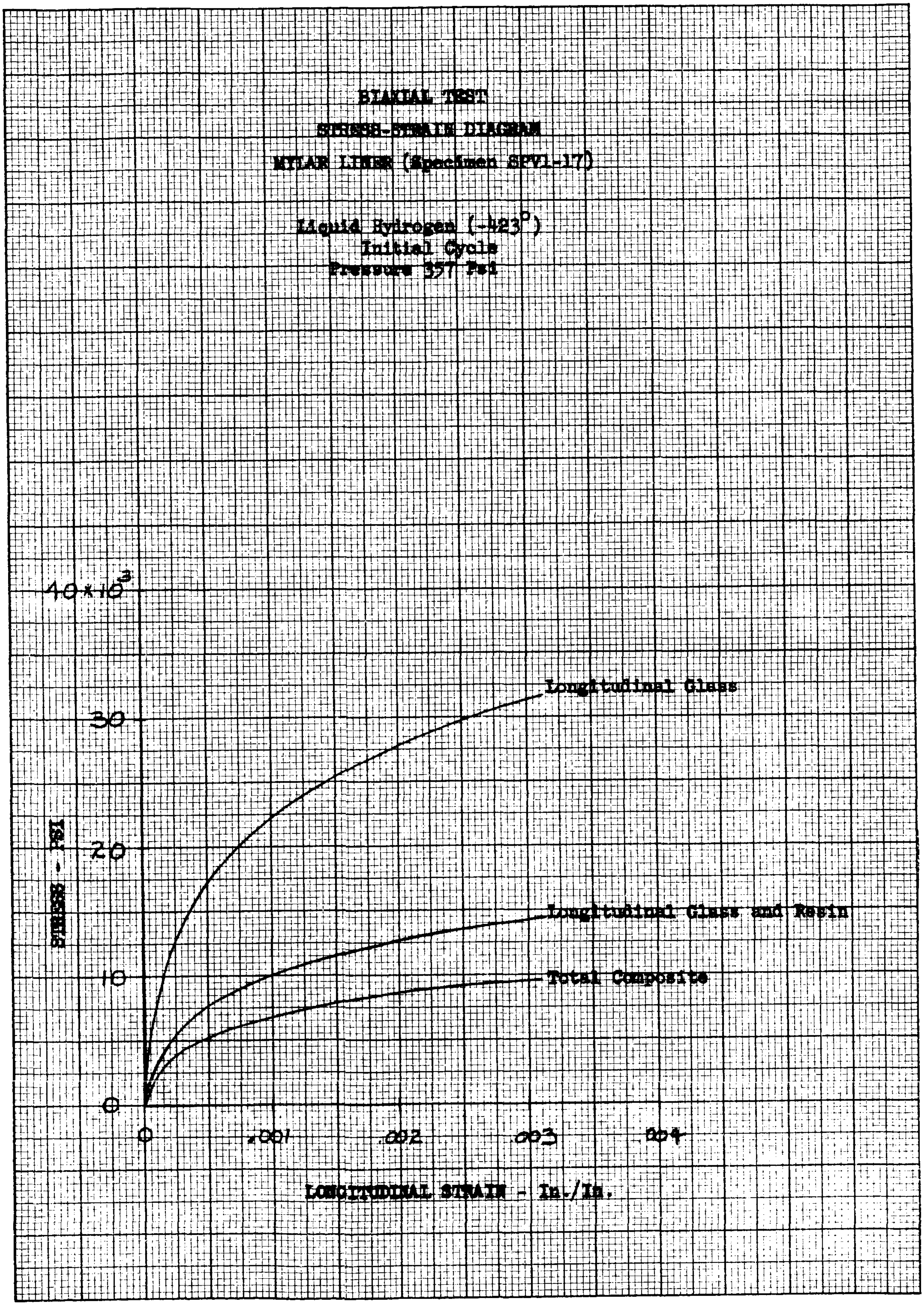

BIAXIAL TEST
STRESS-STRAIN DIAGRAM
MYLAR LINER (Specimen SPV1-17)
Liquid Hydrogen (-423°)
Initial Cycle
Pressure 357 Psi
40×10³
30
20
10
0
STRESS - PSI
Longitudinal Glass
Longitudinal Glass and Resin
Total Composite
0
.001
.002
.003
.004
LONGITUDINAL STRAIN - In./In.

FIGURE 27

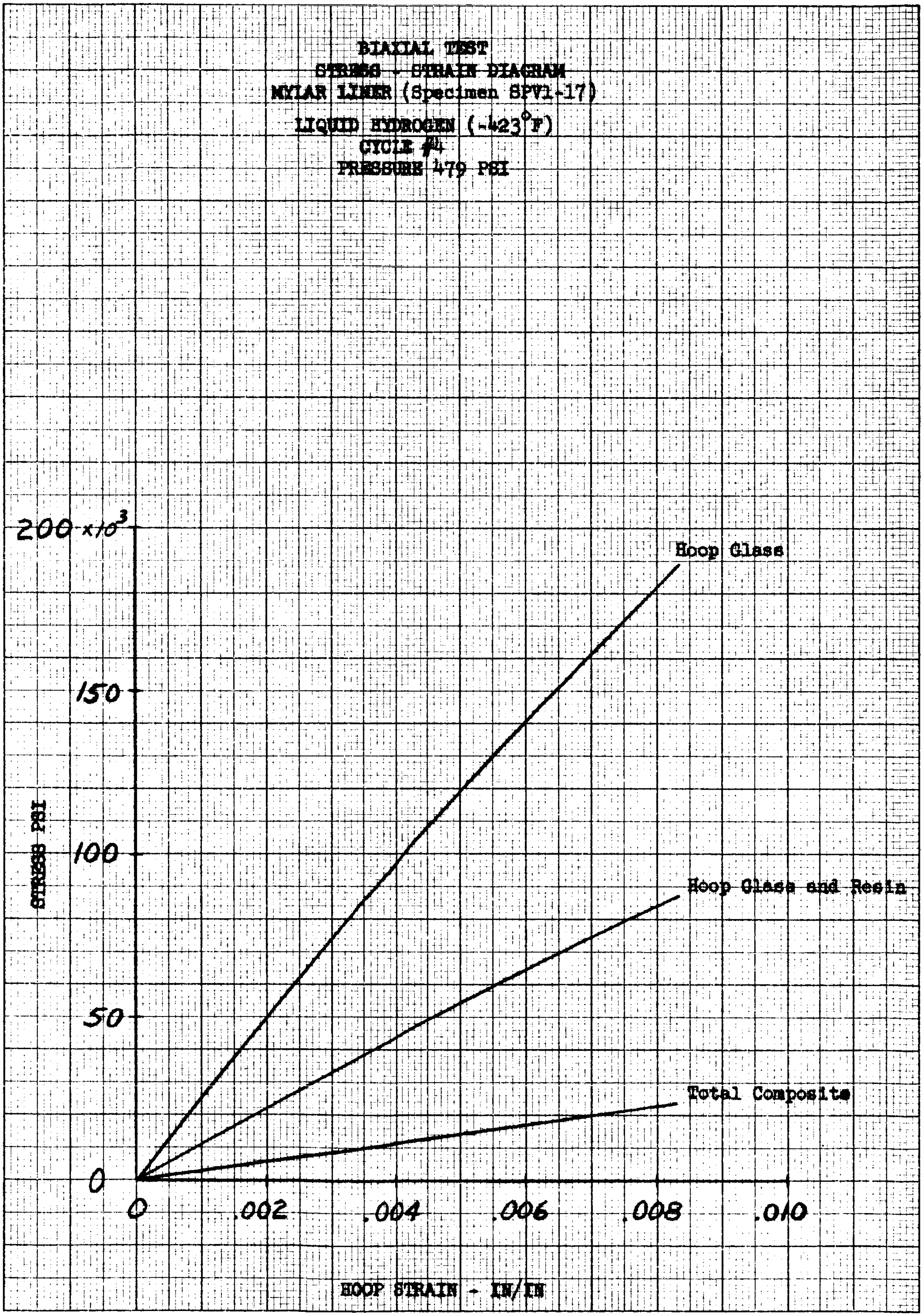

BIAXIAL TEST
STRESS - STRAIN DIAGRAM
MYLAR LINER (Specimen SPV1-17)
LIQUID HYDROGEN (-423°F)
CYCLE #4
PRESSURE 479 PSI
200 x10^3
150
100
50
0
STRESS PSI
Hoop Glass
Hoop Glass and Resin
Total Composite
0
.002
.004
.006
.008
.010
HOOP STRAIN - IN/IN

FIGURE 28

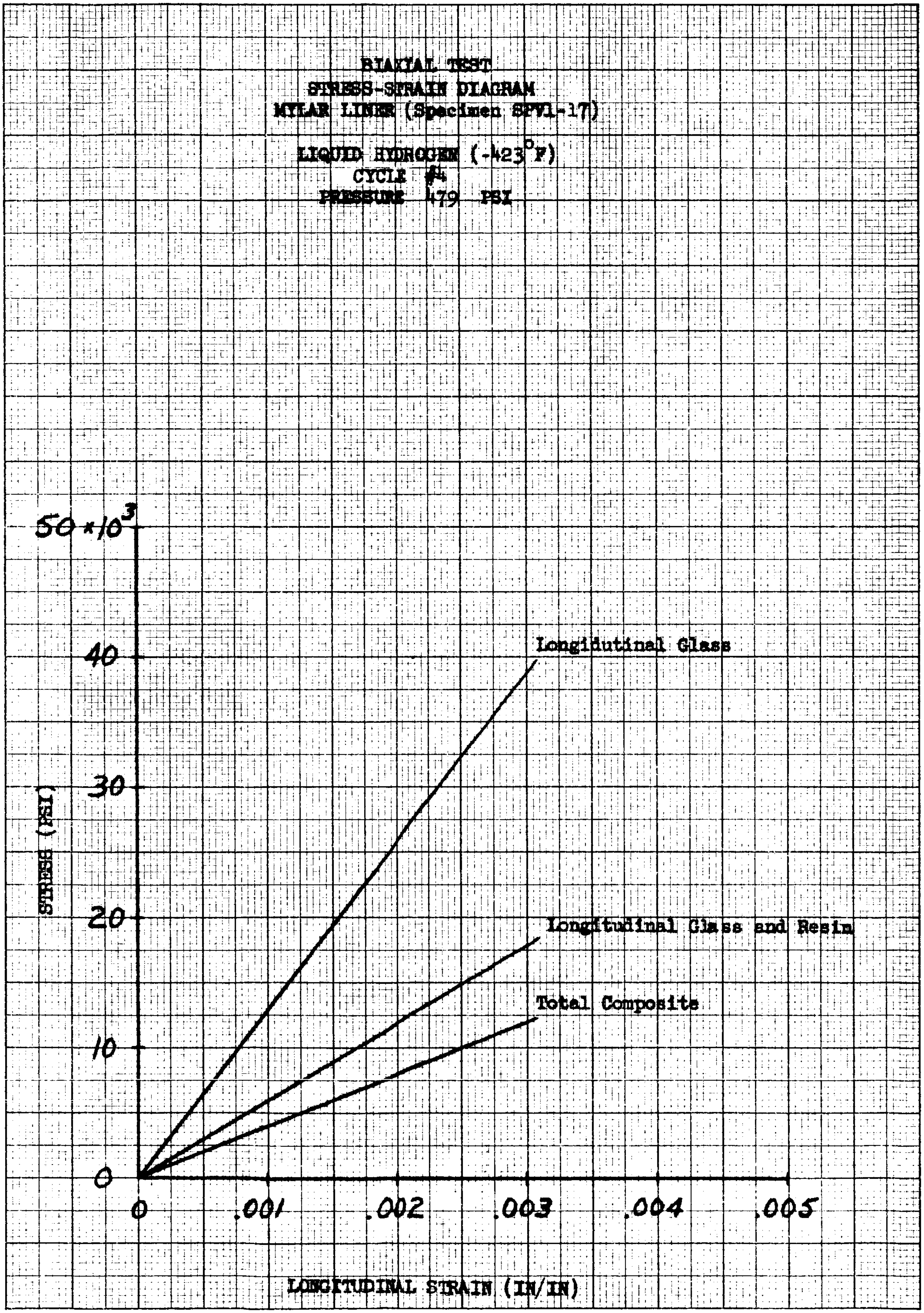

BIAXIAL TEST
STRESS-STRAIN DIAGRAM
MYLAR LINER (Specimen SPV1-17)
LIQUID HYDROGEN (-423°F)
CYCLE #4
PRESSURE 479 PSI
50×10³
40
30
20
10
0
STRESS (PSI)
Longidutinal Glass
Longitudinal Glass and Resin
Total Composite
0
.001
.002
.003
.004
.005
LONGITUDINAL STRAIN (IN/IN)

FIGURE 29

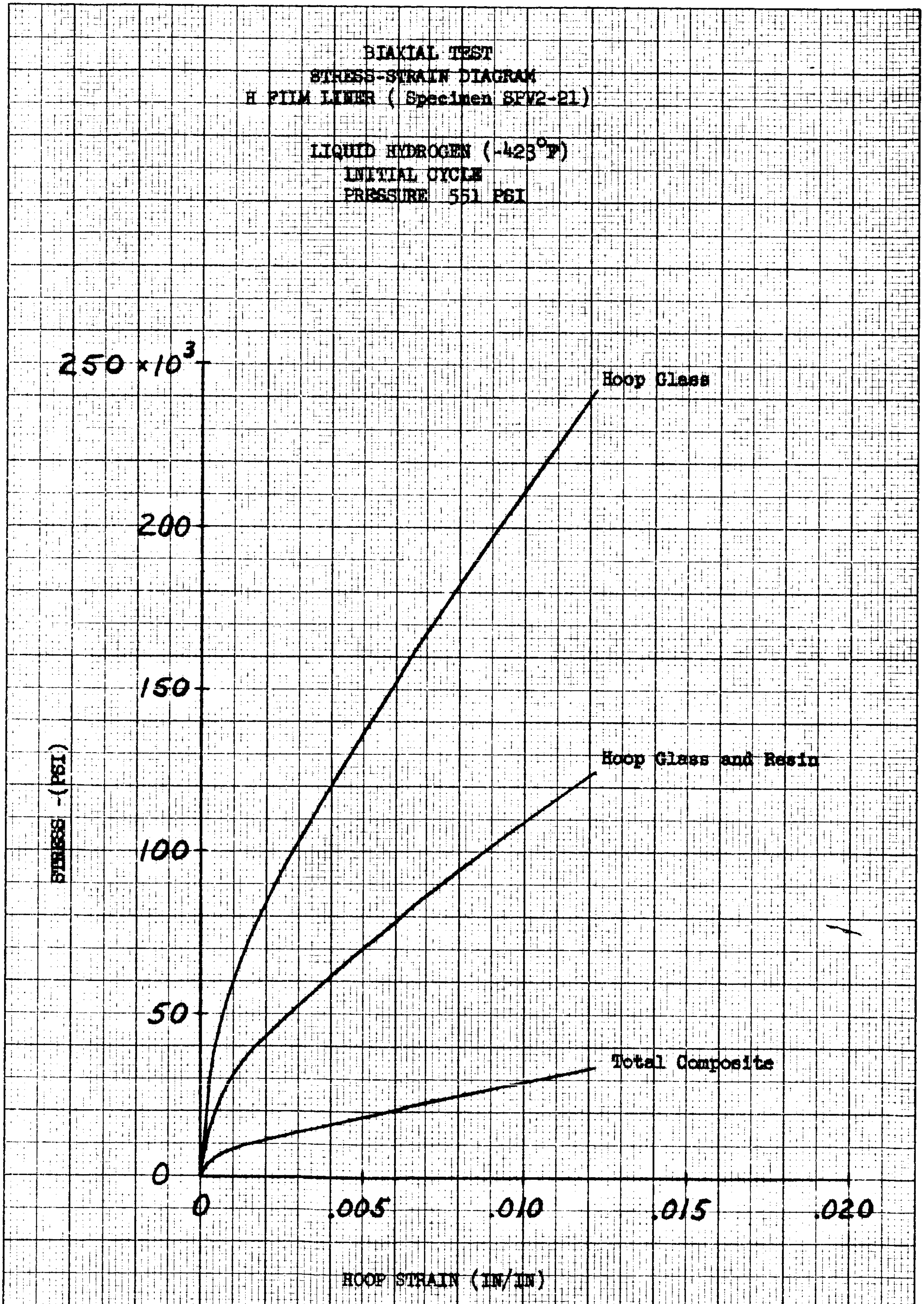

BIAXIAL TEST
STRESS-STRAIN DIAGRAM
H FILM LINER (Specimen SFV2-21)
LIQUID HYDROGEN (-423°F)
INITIAL CYCLE
PRESSURE 551 PSI
250 ×10³
200
150
100
50
0
STRESS -(PSI)
Hoop Glass
Hoop Glass and Resin
Total Composite
0
.005
.010
.015
.020
HOOP STRAIN (IN/IN)

FIGURE 30

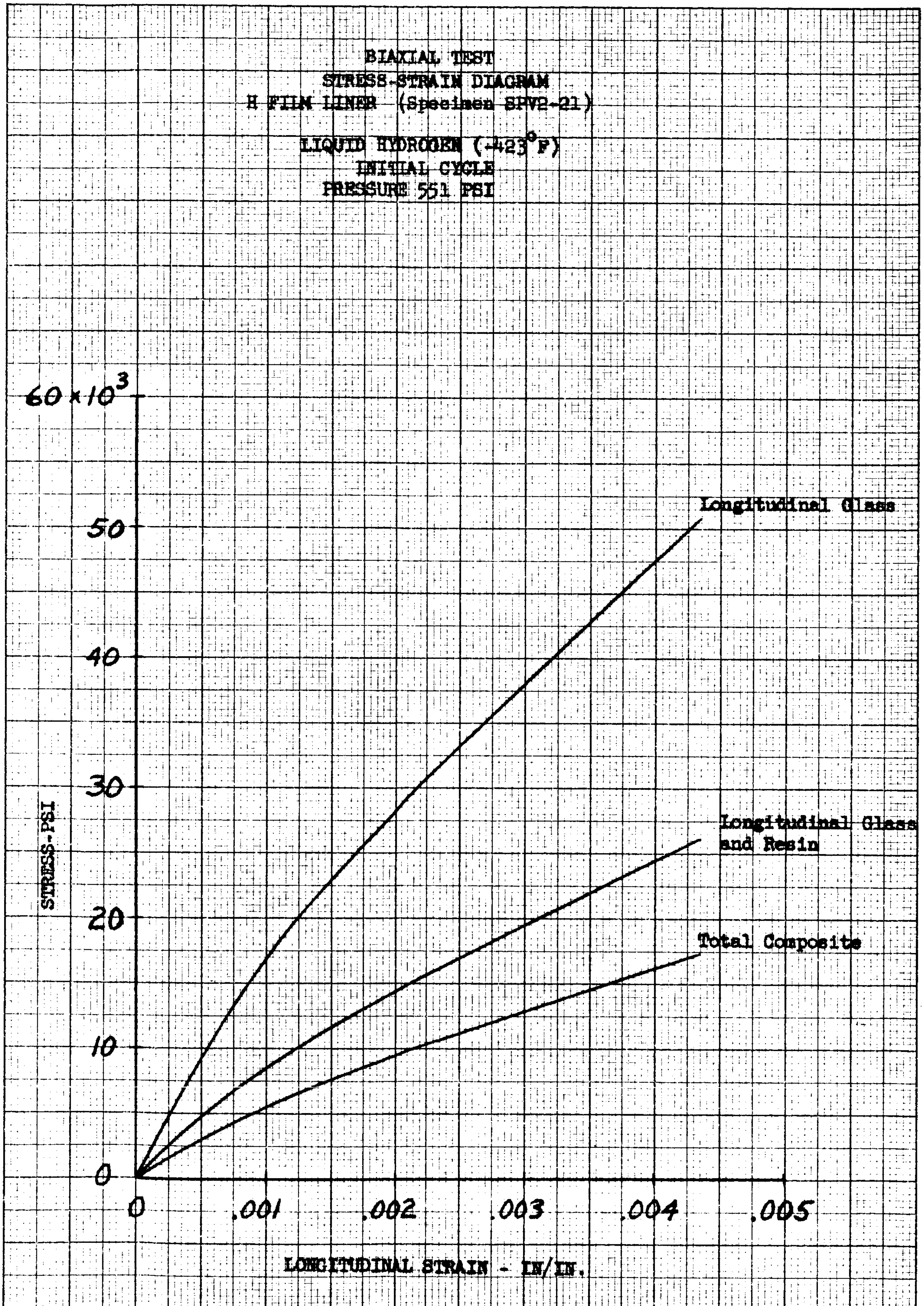

BIAXIAL TEST
STRESS-STRAIN DIAGRAM
H FILM LINER (Specimen SPV2-21)
LIQUID HYDROGEN (-423°F)
INITIAL CYCLE
PRESSURE 551 PSI
60 × 10³
50
40
30
20
10
0
STRESS-PSI
Longitudinal Glass
Longitudinal Glass and Resin
Total Composite
0
.001
.002
.003
.004
.005
LONGITUDINAL STRAIN - IN/IN.

FIGURE 31

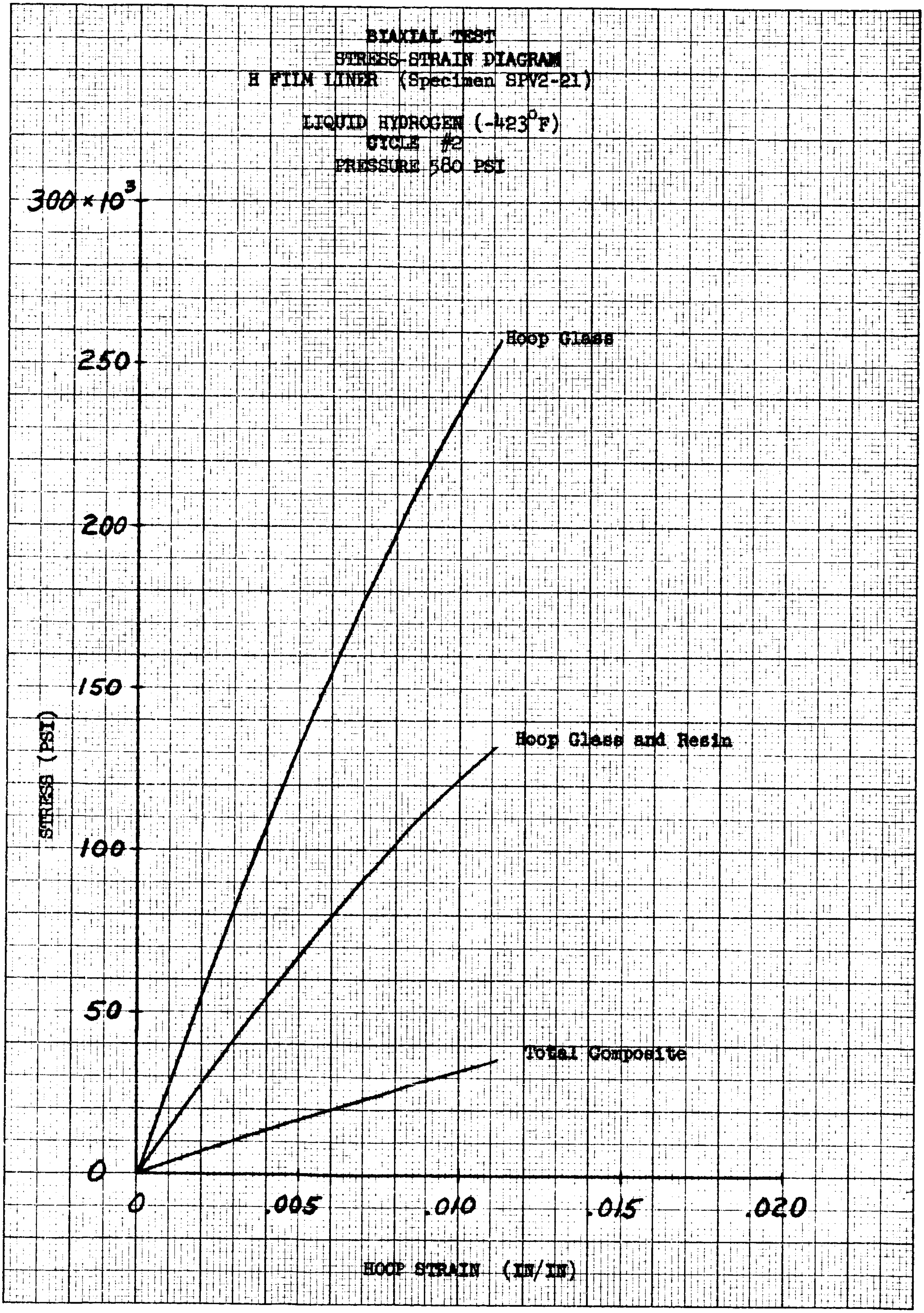

BIAXIAL TEST
STRESS-STRAIN DIAGRAM
H FILM LINER (Specimen SPV2-21)
LIQUID HYDROGEN (-423°F)
CYCLE #2
PRESSURE 580 PSI
300 × 10³
250
200
150
100
50
0
STRESS (PSI)
Hoop Glass
Hoop Glass and Resin
Total Composite
0
.005
.010
.015
.020
HOOP STRAIN (IN/IN)

FIGURE 32

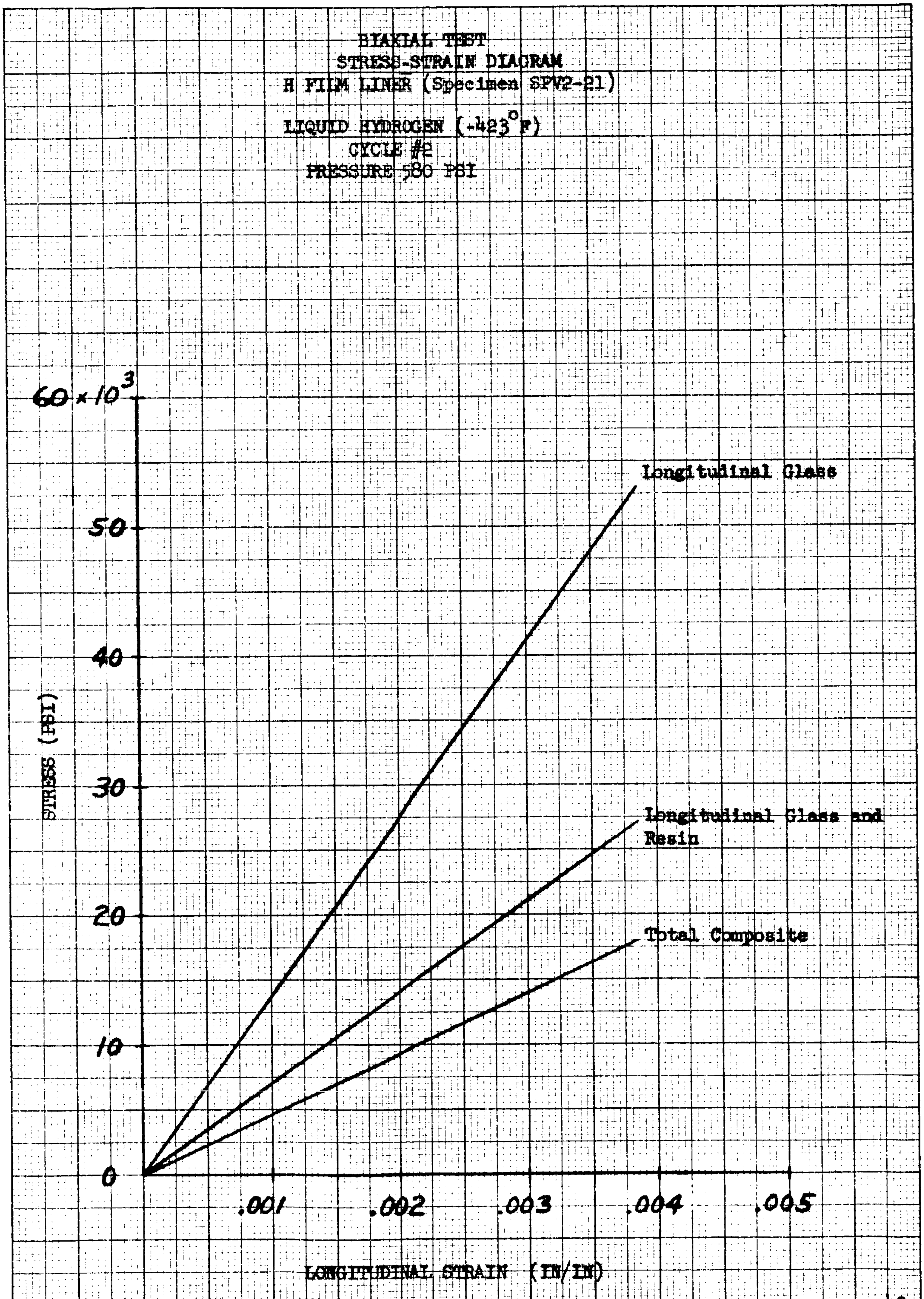

BIAXIAL TEST
STRESS-STRAIN DIAGRAM
H FILM LINER (Specimen SPV2-21)
LIQUID HYDROGEN (-423°F)
CYCLE #2
PRESSURE 580 PSI
60 × 10³
50
40
30
20
10
0
STRESS (PSI)
Longitudinal Glass
Longitudinal Glass and Resin
Total Composite
.001
.002
.003
.004
.005
LONGITUDINAL STRAIN (IN/IN)

FIGURE 33

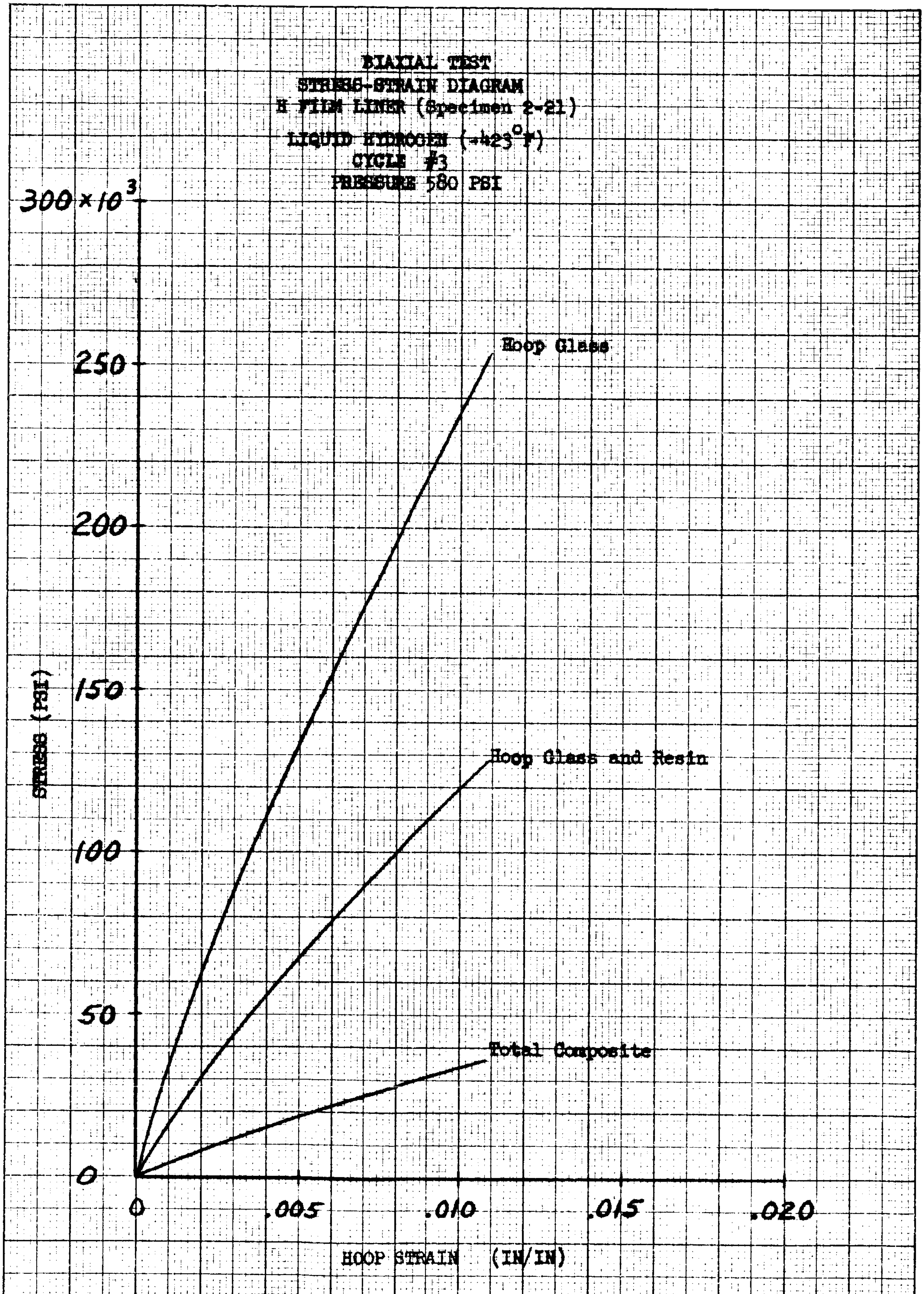

BIAXIAL TEST
STRESS-STRAIN DIAGRAM
H FILM LINER (Specimen 2-21)
LIQUID HYDROGEN (-423°F)
CYCLE #3
PRESSURE 580 PSI
300 × 10³
250
200
150
100
50
0
STRESS (PSI)
Hoop Glass
Hoop Glass and Resin
Total Composite
0
.005
.010
.015
.020
HOOP STRAIN (IN/IN)

FIGURE 34

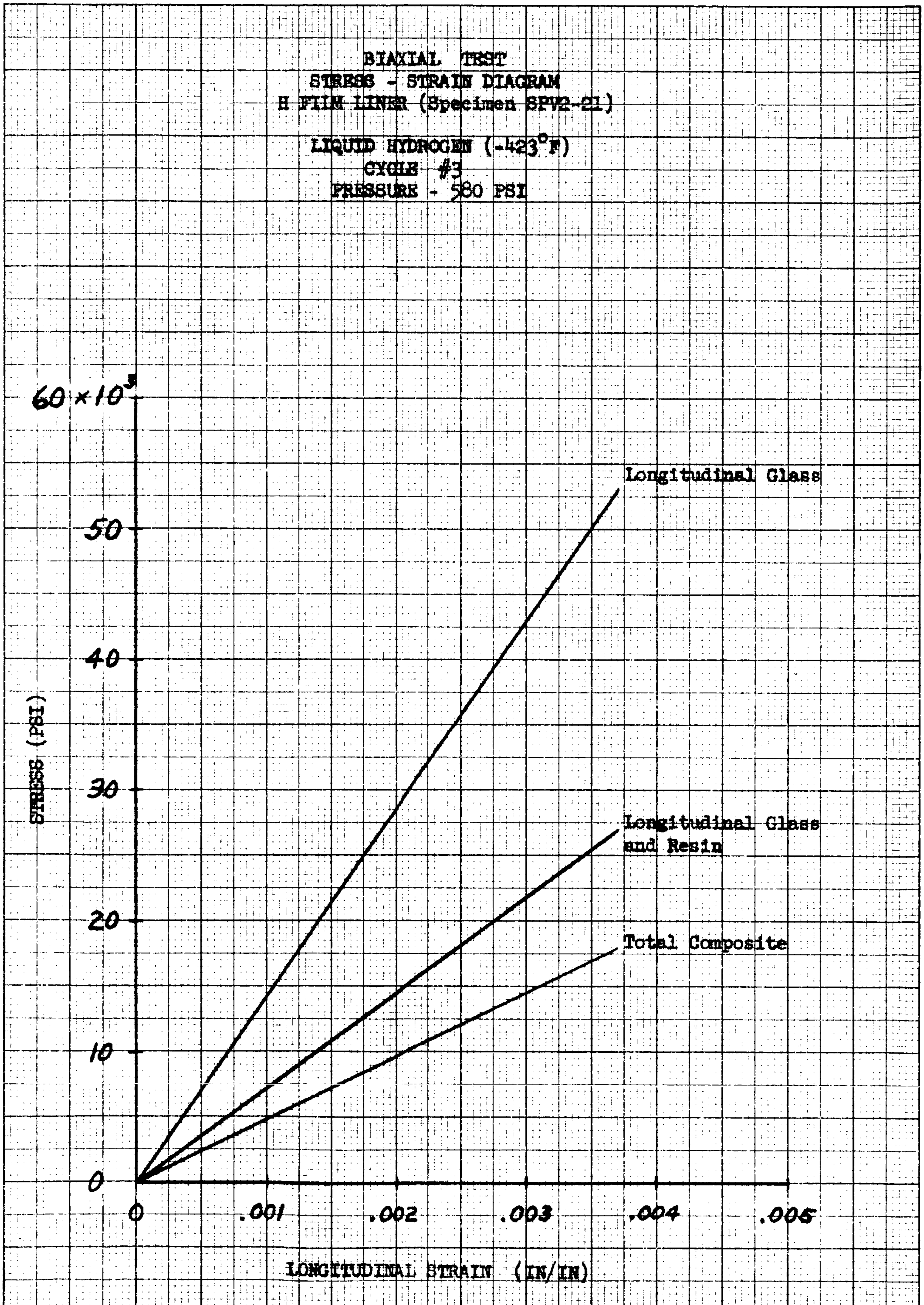

BIAXIAL TEST
STRESS - STRAIN DIAGRAM
H FILM LINER (Specimen SPV2-21)
LIQUID HYDROGEN (-423°F)
CYCLE #3
PRESSURE - 580 PSI
60 × 10³
50
40
30
20
10
0
STRESS (PSI)
Longitudinal Glass
Longitudinal Glass and Resin
Total Composite
0
.001
.002
.003
.004
.005
LONGITUDINAL STRAIN (IN/IN)

FIGURE 35

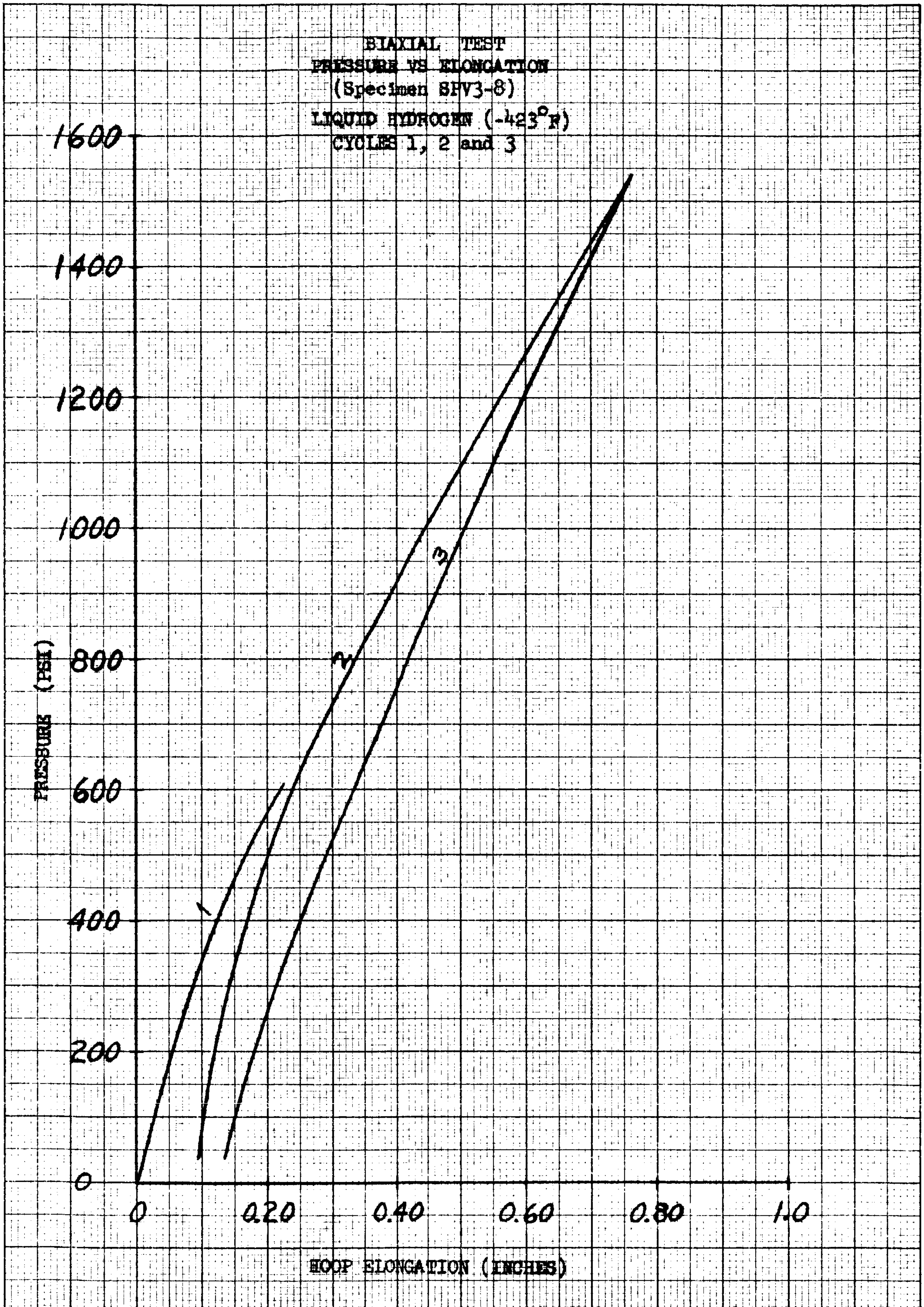


FIGURE 36

K+E 10 X 10 TO THE 1/2 INCH 358T-11 KEUFFEL & ESSER CO.

FIGURE 37

MODEL DATE

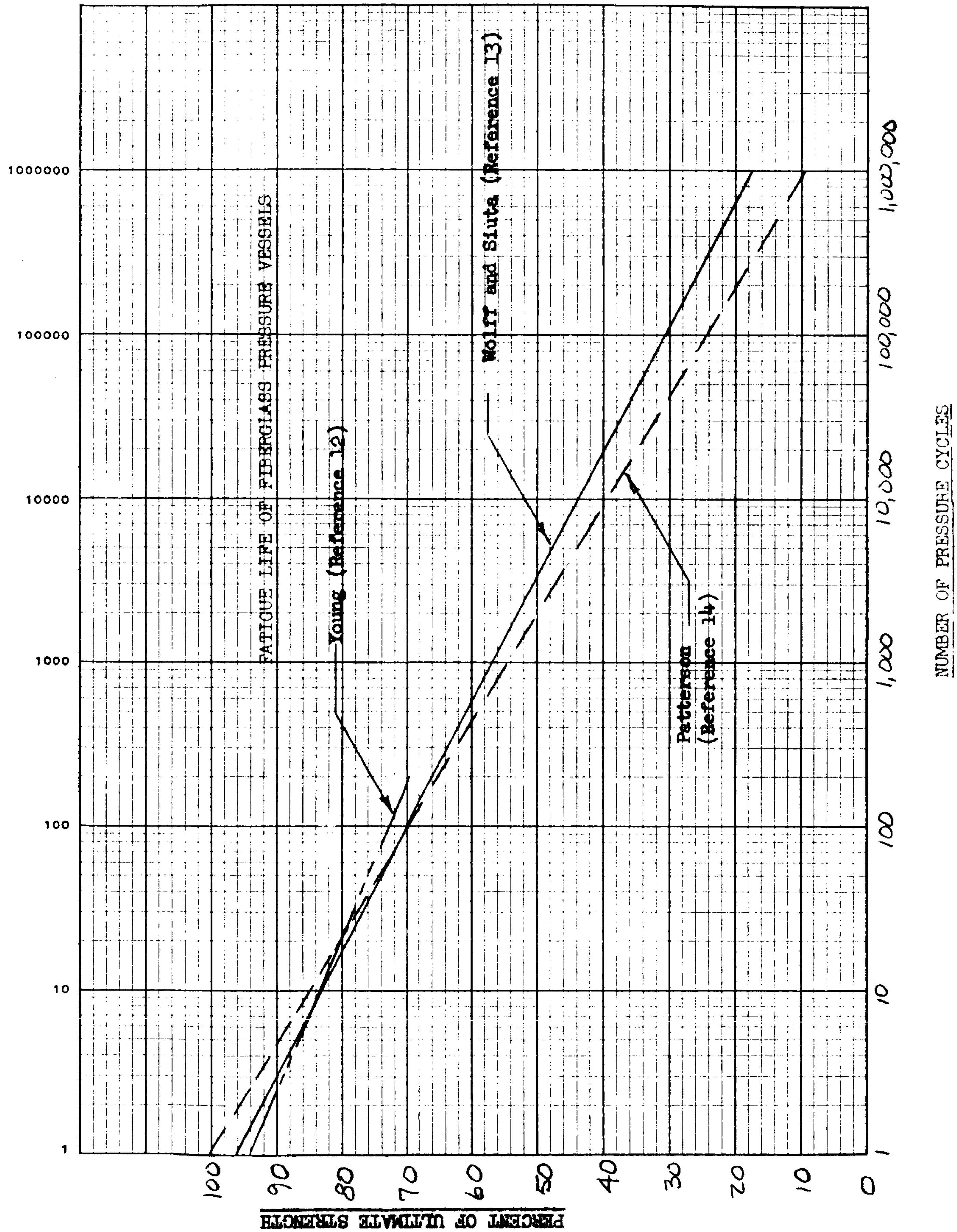


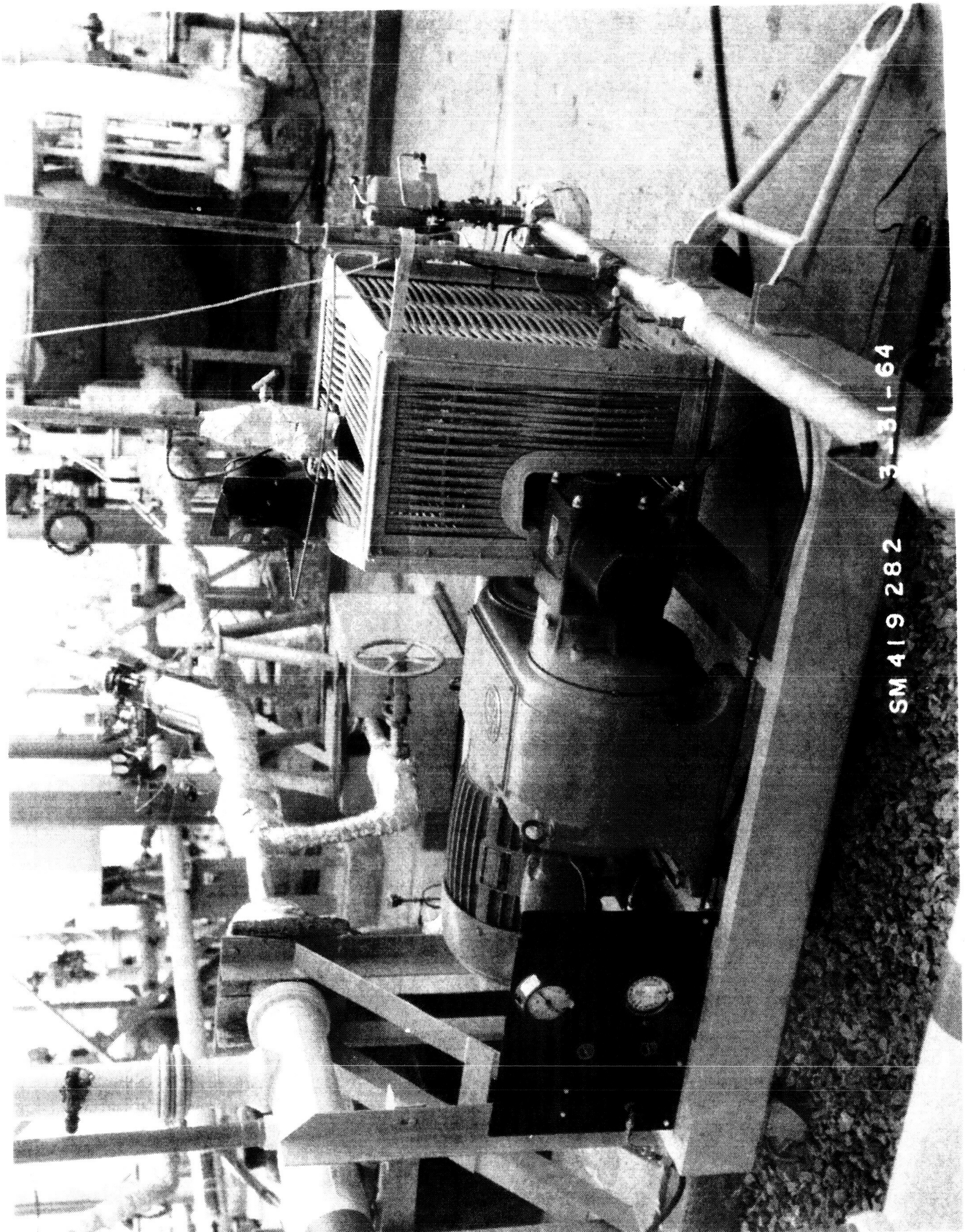
PLATE 96 K·E ALBANENE 195L

To establish a performance standard and choice of electrical resistance strain gages for work with the 18" diameter vessels, an electrodeposited silver lined specimen (SFV2-2) was instrumented with two types of selected electrical resistance strain gages, Type No. S420 and Type No. CXX-620 gages made by the Budd Company. Six gages were installed on the specimen. In addition, the normally used hoop and four longitudinal mechanical deflection gages were installed for correlation of the data.

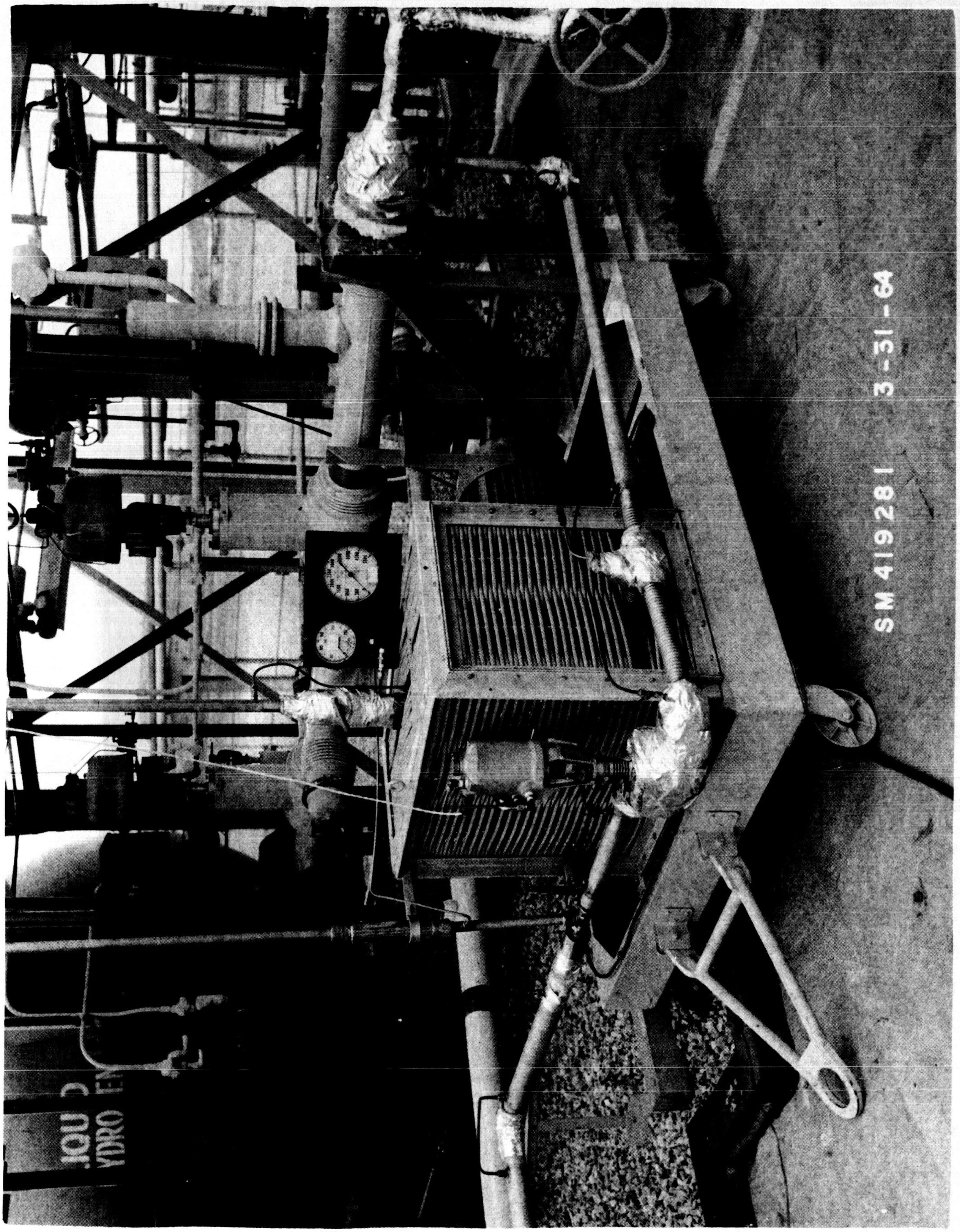
The specimen was pressurized at liquid hydrogen temperature. Four cycles of approximally 249 psi, 245 psi, 582 psi, and 695 psi respectively were made. Liquid hydrogen was used to initially pressurize the specimen on all four cycles; a helium boost was required on the last two cycles. The gages were bonded to the fiber glass structural wall with Narmco 7343/7139 polyurethane adhesive. Examination of oscillograph data indicated complete loss of the gage circuit upon initial pressurization with most of the gages and the remaining gages were erratic in strain indication. Reasonably accurate results were expected to strains of 1% (references 15-17) but such was not the case.

The vacuum chamber which was used for the 7 1/2-inch diamater by 20-inch long filament-wound test cylinders will also be used for testing the 18-inch diameter by 24-inch long pressure vessels. Rework was required, (1) to permit proper location of new deflection gage points, and (2) to better seal all interior fittings in order to hold the vacuum for insulation purposes and identification of vessel failure. In addition, the transfer facilities have been reworked ard vacuum jacketed. Installed cryogenic pump is shown in figures 38 and 39. The test schematic is shown in figure 40.

The 7 1/2-inch diameter cylinders are sealed, using a mechanical indentation technique to lock the liner, backed by the fiberglass flange, against the test plates. This method has sealed these specimens sufficiently to perform the tests required to date. One disadvantage of this seal is that when the mechanical seal is effected, the liner is deformed. The sealed specimen can not be resealed reliably if the seal should be removed for an internal inspection. A more important disadvantage is that leakage causes loss of chamber vacuum and as a consequence both insulation and chamber pressure indications are lost; loss of insulation causes excessive boil-off in the test



Cryogenic Pump - Vacuum - jacketed transfer lines
and vent stacks
FIGURE 38



Cryogenic Pump - Wire Mesh Protection cage and Mobil cart installation

FIGURE 39

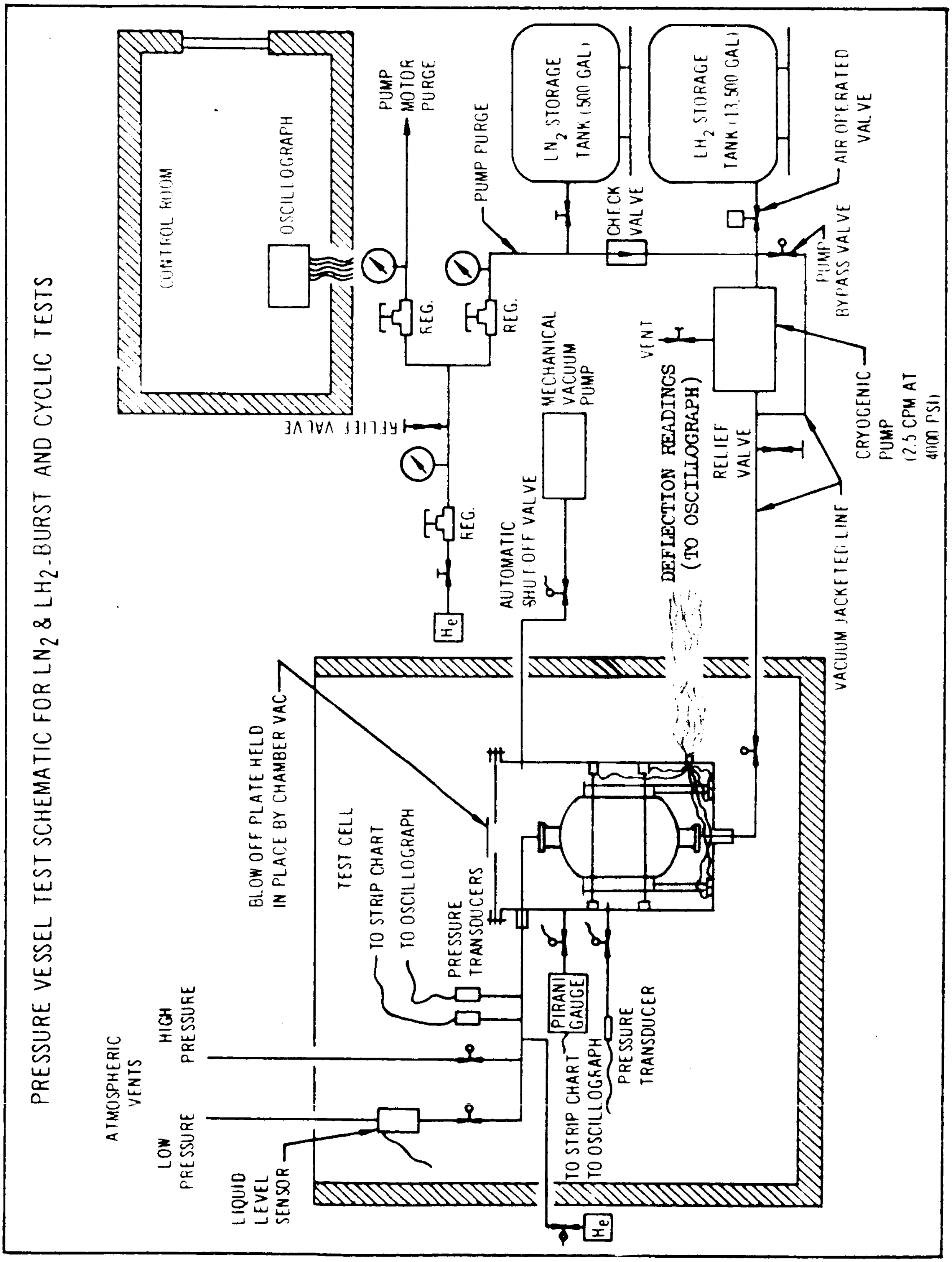

PRESSURE VESSEL TEST SCHEMATIC FOR LN2 & LH2-BURST AND CYCLIC TESTS
CONTROL ROOM
OSCILLOGRAPH
PUMP MOTOR PURGE
PUMP PURGE
LN2 STORAGE TANK (500 GAL)
LH2 STORAGE TANK (13,500 GAL)
CHECK VALVE
AIR OPERATED VALVE
PUMP BYPASS VALVE
REG.
REG.
REG.
RELIEF VALVE
MECHANICAL VACUUM PUMP
VENT
DEFLECTION READINGS (TO OSCILLOGRAPH)
CRYOGENIC PUMP (2.5 CPM AT 4000 PSI)
RELIEF VALVE
VACUUM JACKETED LINE
AUTOMATIC SHUT-OFF VALVE
He
BLOW OFF PLATE HELD IN PLACE BY CHAMBER VAC
TEST CELL
TO STRIP CHART
TO OSCILLOGRAPH
PRESSURE TRANSDUCERS
ATMOSPHERIC VENTS
LOW PRESSURE
HIGH PRESSURE
LIQUID LEVEL SENSOR
PIRANI GAUGE
PRESSURE TRANSDUCER
TO STRIP CHART
TO OSCILLOGRAPH
He

FIGURE 40

specimen and associated piping with attendant subsequent chilldown and pressurization problems, and loss of chamber pressure indication makes it impossible to determine the point of liner/shell failure for anything other than catastrophic failure.

To provide a better seal for the 18-inch diameter vessels, discussions were held with personnel at the Cryogenic Engineering Laboratory of the National Bureau of Standards. Their recommendations have led to the evaluation of a sleeve confined O-ring design (figure 41). Resealing is possible with only the insertion of a new eleastomer O-ring. Testing has been done with a 3" diameter cylinder, which was fabricated exactly as the integral vessel end fittings. Various size O-rings have been tested at -320°F. A fabrication defect in the test cylinder has prevented making a positive selection at the present time.

2.3 18-INCH DIAMETER VESSEL DESIGN AND FABRICATION

2.3.1 Design

The geodesic-isotensoid dome shape has been determined as outlined by Read (reference 18) and additional information for geometrical properties has been detailed by Stone (reference 19).

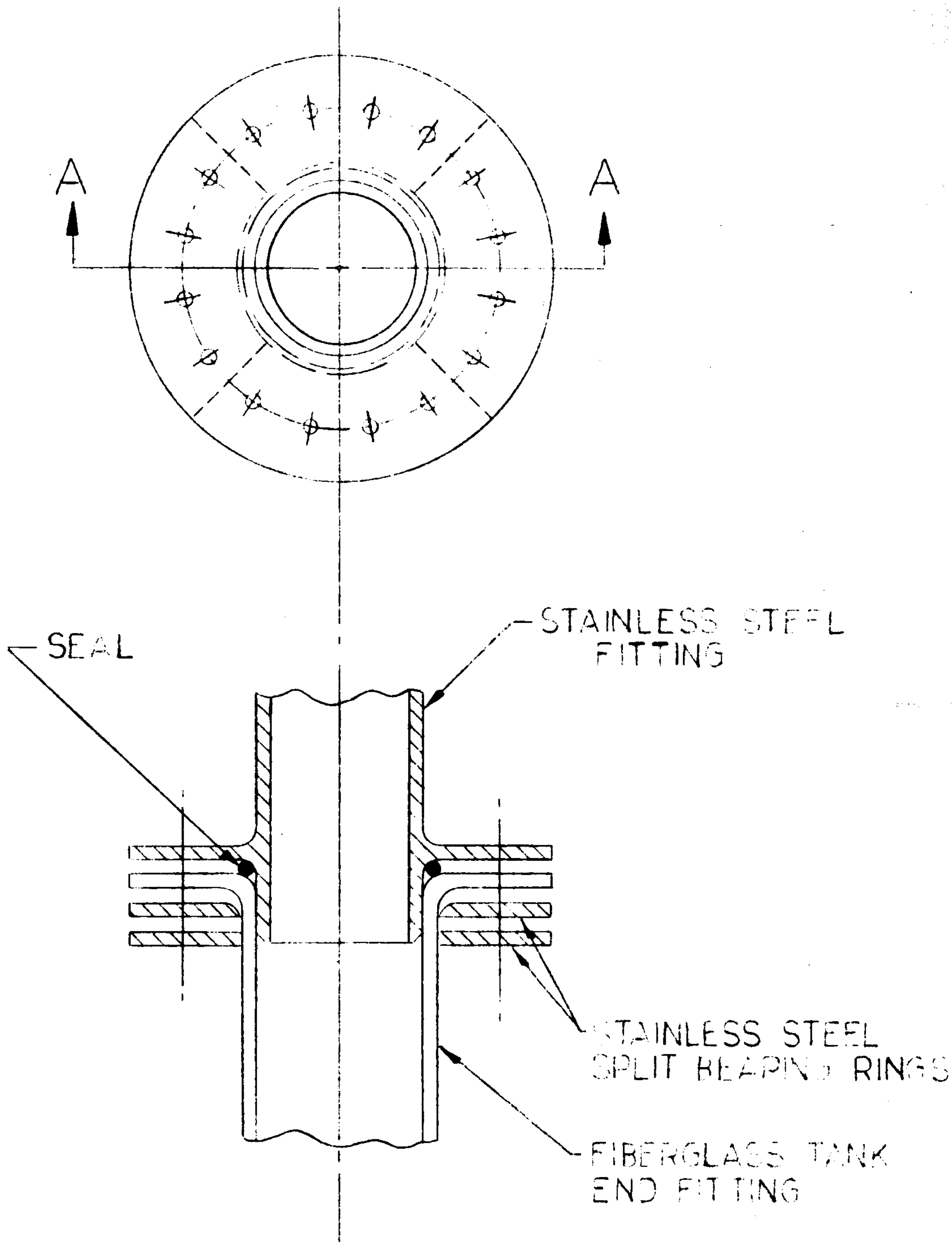
A rational method for selecting filament strength and calculating wall strengths has been reported by Darms (reference 20) and has been followed for this program.

The integral end fitting design has been governed by providing a smooth transition between the shell and fitting and the need to put the tapered edge of the fitting beyond the dome inflection point.

2.3.2 Tooling

2.3.2.1 Handling Fixtures

The handling fixtures for transporting the mandrel within the plant were adapted from existing fixtures. Two shipping containers used to transport the mandrel to the Vendor have been designed and fabricated. Three small foam-cushioned cradles used to handle and transport the completed vessel from the



SECTION A-A

FIBERGLASS TANK END FITTING SEAL

FIG. 41

fabrication area to the test area have been built. All handling, storage, and transportation fixtures are available.

2.3.2.2 Salt Mandrel Hardware

Originally, three mandrel shaft units were fabricated for this program by modifying an existing 18" diameter by 24" long filament-winding salt mandrel shaft design. The mandrel shaft unit consists of an outer shaft which slides on an inner shaft.

End-fitting mandrels were designed and fabricated to slip over the ends of the shaft and butt against the salt to provide a continuous surface for the deposition of the electroformed liner.

The interface between the end-fitting mandrel and mandrel shaft is sealed with zinc chromate. At the joint of the salt and metal mandrels, an acetone based body putty (Mason's surfacer) is used to fill the joint. This body putty is hand-sanded smooth to shape.

On the first attempt to seal the outer shaft, inner shaft, salt casting, and end fitting mandrel combination, the end fitting mandrels, which are mounted directly to the inner shaft, moved at the salt interface whenever the inner shaft moved in flexure. This damaged the seal at this joint, and leaks became evident during the water soak tests. Therefore, the shafts were reworked to provide a stiffer support. This was accomplished by lengthening the outer shaft to the length of the inner shaft. The new shafts were completed within a week.

Three sets of outer and inner mandrel shafts and end-fitting mandrels have been completed. The shafts have been designed to fit salt slush molding fixture adapters, the sealing fixture adapters, and the filament winding machine adapters. All the adapters are available for this use.

2.3.2.3 Salt Mandrel Casting

The slush-molding equipment used to cast salt mandrels is used without modification.

Mandrel Serial #002: Cracked during initial fabrication. This mandrel was cast only. No machining was accomplished.

Mandrel Serial #003: This mandrel was cast while investigations were continuing on #001. Due to the redesign of the shaft, and apparent inability of #003 to work, the salt was removed from the steel shaft.

Mandrel Serial #004: This will be the first mandrel cast on the redesigned stiff shafting.

2.3.3 Vessel Fabrication

A wooden practice mandrel machined to the shape of a lined salt mandrel has been prepared with scribed geodesic paths. This mandrel has been used to develop the proper shape for the cams used to control the winding arm.

Also tested with the wooden mandrel was an advanced impregnation-tension device installed on the winding machine. The tension is not applied to the rovings until after impregnation. Impregnation is done in a small dip tank.

Several complete helical cycles were wound on the wooden mandrel using four spools of 12-end S994/HTS Fiberglas, and ERLA 0510/ZZL0803 room temperature curing epoxy resin. These wraps were allowed to cure and inspected closely. As a result of these exercises, the new and old winding equipment is compatible and the operator trained.

2.3.2.4 Salt Mandrel Sealing

Due to the selection of a metallic liner, the problem of effective salt sealing had to be solved. The previous Douglas IR&D program had shown the feasibility of the process, but better results had to be obtained for a quality product. Using small salt samples, an effective seal has been made. Smoothing of surface pits with a high temperature wax has been excellently accomplished. The sealing investigation was made with both a spray and dip operation of (1) PVC (polyvinylchloride), and (2) an acrylic lacquer. The spray method did not provide an adequate seal. The dip method worked with both the PVC and the acrylic lacquer, when subjected to the test environment--185°F aqueous bath for two hours. The acrylic lacquer is used for the 18-inch diameter vessels since it bonds to the salt better than the PVC. A full size 18" diameter salt mandrel was used to optimize the dip coat operation.

After being machined the mandrel was dip coated with a 50/50 acrylic/acetone mixture. This was washed off and a 20/80 acrylic/acetone coat was applied by dipping 1/2 way and rotating by hand. This deposited a coat approximately 1 mil thick with 4 mil waves. After installing a motor to rotate the mandrel and exercising precise rotation control, the waves disappeared. The rotation used was in the range of 12 RPM. This coat was left on and two 25/75 acrylic/acetone dip coatings were applied. This applied a seal coat of approximately 3 to 5 mils. This is the procedure used to seal the salt mandrel. A small dip tank is available and used in conjunction with the slush molding fixture. A water submersion test is the final acceptance test for the sealed salt mandrel assembly.

2.3.2.5 Fabrication of Salt Mandrels

Mandrel Serial #001: The mandrel was cast and machined to size. It was sealed with 3 dip coats of an acrylic/acetone mixture which built up a 5 mil thickness. Due to the stiffness problem mentioned in 3.2.2, and due to excessive handling, while testing and investigating the problem, the mandrel developed excessive pinholes and leaks. The mandrel had been exposed to several thermal cycles from storage in the oven and several water bath seal tests. Mandrel #001 was scrapped.

SECTION 3

PHASE II - FABRICATION OF SMALL-SCALE PRESSURE VESSELS

The objective of Phase II is to fabricate 20 small-scale fiber glass filament-wound pressure vessels for cyclic and burst testing at -320°F and -423°F. The tanks will be designed for a nominal proof pressure of 500 psi and a nominal burst pressure of 600 psi. To provide the desired mode of failure, the design may possibly have to be revised during the fabrication period; therefore, the fabrication is set up on an intermittent production schedule.

SECTION 4

PHASE III - STRENGTH AND CYCLING TESTS AT -320°F

The objective of Phase III is to determine the ultimate strength and cycling capability of 10 small-scale fiber glass filament-wound vessels at liquid nitrogen temperature. In any pressure vessel design, it is necessary to know the maximum pressure the vessel can withstand and how many cyclic loadings can be applied at a given stress level. Ultimate pressure capability is self-explanatory. Cycling tests are important in any evaluation of filament-wound pressure vessels with liners because differences in strain characteristics of the resin/fiber glass system and the liner material may cause ultimate failure of the system.

Girth and longitudinal strain measurements are necessary for evaluating the tank and liner during cycling tests and for collecting meaningful data. Of equal importance is the measurement of gas content into the burst chamber during the tests, because this measurement will be an indication of liner breakdown under cyclic loads.

Phases III and IV of the proposed program call for testing the 18-inch diameter x 24-inch long vessels manufactured in Phase II. Liquid nitrogen is to be used as the pressurizing fluid in Phase III, while liquid hydrogen is used in Phase IV; other than this, the two phases are identical in proof pressures, pressure cycling requirements, and data to be obtained.

At this point of the project, work in this phase is that reported under pertinent discussions in Section 2.2.3.

SECTION 5

PHASE IV - STRENGTH AND CYCLING TESTS AT -423°F

The objective of Phase IV is to determine the effect of -423°F on the ultimate strength and cycling capability of 10 small-scale filament-wound fiber glass tanks. The data sought here is the same as in Phase III.

As stated in Section 4, the only difference between the test procedure of Phase III and Phase IV is that Phase IV uses liquid hydrogen rather than liquid nitrogen. The test set-up and instrumentation will be used without any changes.

At this point of the project, work in this phase is that reported under pertinent discussions in Section 2.2.3.

SECTION 6

RELIABILITY/QUALITY ASSURANCE

6.1 VENDOR CONTROL

A meeting was held with the electrodeposition vendor early in January. The vendor was very cooperative and gave every indication that he will comply with the project requirements. Further informal contacts with the vendor showed that he considerably improved his quality control procedures.

In March, Quality Engineering performed a follow-up quality standard and process control survey of the vendor. This survey indicated that the electrodedeposition vendor has met the requirements for this contract.

Due to previous scatter in the reported elongation values of batches of electrodeposited nickel by the vendor and the data obtained from coupons of the same batch by Douglas, a close examination of the test procedures of both companies was made. As a result of this examination, Douglas testing of a sample batch of electrodeposited nickel using Electroforms Inc. size and shape tensile specimen, and careful machining of the specimen to size, produced data in the same magnitude as Electroforms Inc. data; i.e.,

18% Elongation (Douglas)
17% Elongation (Electroforms Inc.)

102,000 psi Ultimate (Douglas)
100,000 psi Ultimate (Electroforms Inc.)

A meeting was held with the vendor, representatives of Douglas, and the NASA Project Manager in March to further inform the vendor of the interest in his quality and process control.

6.2 MANUFACTURING INSPECTION

Close liaison between manufacturing, inspection, and engineering has resulted in realistic and detailed inspection check points during the fabrication of the 18" diameter filament-wound pressure vessel. These inspections will be recorded on the Fabrication Outline (FO) for each vessel and become a part of the permanent record.

SECTION 7

OUTLINE OF WORK FOR NEXT QUARTER

The work expected to be completed during the third quarter of the program is listed below:

1. Two 18-inch diameter pressure vessels for Phase I will be fabricated and tested at -423°F.

2. Phase II fabrication will commence.

3. Phase III and Phase IV testing will commence.

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